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(U) DEVELOPMENT OF HIGH-ENERGY  
METALLIZED PROPELLANTS

Aerojet-General Corporation  
Sacramento, California

TECHNICAL REPORT AFRPL-TR-66-230

September 1966

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FOREWORD

This is the final report on the Development of High-Energy Metalized Propellants covering the period from 1 May 1965 through 31 August 1966, and is submitted to the Rocket Propulsion Laboratory in partial fulfillment of Contract AF 04(611)-10783. Publication of this report does not constitute Air Force approval of the report's findings or conclusions.

The investigations were conducted by the Advanced Propulsion Research Department, Research and Technology Operations, Aerojet-General Corporation, Sacramento, California. This program was conducted for the Liquid Rocket Division of the United States Air Force Rocket Propulsion Laboratory, Edwards, California. Lt. A. Tatkenhorst was the Project Officer for the Air Force.

This report was prepared by the staff of Liquid Propellant Research and Development under the direction of W. R. Fish, Project Manager. J. K. Suder and A. J. Aitken were Associate Project Managers for the propellant and engineering investigations, respectively. Contributors to the report also include R. E. Anderson, J. J. Bost, R. H. Globus, R. V. Wright, and E. M. Vander Wall of Liquid Propellant Research and Development; and P. J. Petrozzi and R. M. Thomas of Aerophysics.

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### CONFIDENTIAL ABSTRACT

(C)  $\text{N}_2\text{O}_4$ /Beryllizine-21(I) and 98%  $\text{H}_2\text{O}_2$ /Beryllizine-28(II), were investigated in a 1000-lb thrust test assembly operated at 500-1100 psia chamber pressure to develop techniques for ignition and efficient combustion of the metal. Ignition was achieved by the use of an injector concept that involves a two-part design. Each of the two types of injectors designed for the investigations contained a central hot-core section within a second, peripheral injection area. By operating the hot-core at a high mixture ratio, the temperature of combustion of the oxidizer with the hydrazine in the fuel was high enough to ignite the suspended beryllium. The peripheral section of the injector was operated at such a mixture ratio as to produce an overall ratio favorable to high efficiency of combustion. For (I) the highest specific impulse obtained was 321 sec (86.3% of theory) at a mixture ratio of 0.71 at 1100 psia chamber pressure. In the case of (II) the highest specific impulse obtained was 344 sec (90.3% of theory) at a mixture ratio of 1.1 at 977 psia chamber pressure.

(C) The flow properties of Beryllizine-21 were also investigated. The ASTM-D-1092 capillary viscometer was used to characterize flow in the laminar region. Alumizine-43G was also examined for comparison purposes and the data show that these two fuels have similar flow properties. When Beryllizine-21 was flowed in 1/4-in. dia stainless steel test sections, the results were consistent with those obtained with the capillary viscometer. Flow loop tests with Beryllizine-21 in the turbulent region showed that in this case also the flow properties are nearly the same as those of Alumizine-43G when the two fuels have comparable yield stress values.

(C) The fuels used in the engineering investigations were prototype formulations made with materials and processes that were state-of-the-art at the beginning of the program. These fuels had desirable physical and rheological properties, but they were not suitable for long-term storage due to the chemical incompatibility of the components. Propellant investigations were conducted concurrently with the engineering investigations to determine the sources of incompatibility and to develop and characterize final formulations that were mechanically and chemically stable during long periods of storage. The chemical compatibility investigations showed that, in general, the purest beryllium powders were the most compatible with hydrazine. The impurities that were most suspect as sources of evolved gas were beryllium carbide and magnesium. The water-treatment of chromium-coated powders, particularly those with low contents of impurities, was shown to be the most effective of the methods investigated for the reduction of gas evolution. In addition, it was shown that the Carbopol-940 used to gel the prototype formulations played an important role in the gas-producing chemical reactions. On the other hand, certain Aerojet-developed derivatives of polyacrylic acid were effective gelling agents for Beryllizine that did not affect the chemical stability of the fuel. By combining the use of the new gelling agents with the water-treatment of the beryllium powder, the formulation of chemically stable Beryllizine fuels was made possible.

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Report AFRPL-TR-66-230

iv

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Report AFRPL-TR-66-230

## TABLE OF CONTENTS

	<u>Page</u>
I. Introduction	1
II. Summary	4
III. Technical Discussion	9
A. Chemical Laboratory Investigations	9
1. Objectives	9
2. Criteria Survey and Preparation of Specification	9
3. Propellant Formulation and Characterization	10
a. Compatibility of Propellant Components	10
b. Effect of Beryllium Powders on Gel Formation	38
c. Development of Prototype Fuels	42
d. Survey of Test Methods	57
e. Quality Control Analyses on Prototype Fuels	61
B. Engineering Investigations	64
1. Flow Characterization	64
a. Flow-Loop System Design and Construction	64
b. Data Reduction Techniques	66
c. Test Program	76
2. Ignition and Combustion Technology	89
a. Engineering	92
b. Fabrication	113
c. Test Program	116
d. Results of Testing	152
C. Propellant Handling and Operating Procedures	157
IV. Conclusions and Recommendations	158
A. Conclusions	158
B. Recommendations	160
V. References	162

▼  
UNCLASSIFIED

# UNCLASSIFIED

Report AFRPL-TR-66-230

## APPENDIXES

<u>Appendix</u>		<u>Page</u>
A	Beryllizine Fuel Criteria	165
B	Survey of Vibrations and Acceleration Conditions	179
C	Tentative Military Specification, Propellant, Beryllizine	189
D	Laboratory Processing Procedure for Beryllizine	225

# CONFIDENTIAL

Report AFRPL-TR-66-230

## LIST OF TABLES

<u>Table</u>		<u>Page</u>
I	Composition of Selected Beryllium Powders (U)	14
II	Compatibility of Hydrazine with Untreated Beryllium Powders (U)	15,16
III	Compatibility of Hydrazine with Beryllium Powders Treated with 5% Nitric Acid (C)	17
IV	Compatibility of Hydrazine with Beryllium Powders Treated with 30% Hydrogen Peroxide (C)	18
V	Compatibility of Hydrazine with Beryllium Powders Treated with Hydrazine (C)	19
VI	Compatibility of Hydrazine with Beryllium Powders Treated with Alcoholic Potassium Hydroxide (U)	21,22
VII	Comparative Gas Evolution Rates of Hydrazine Containing Caustic Treated and Untreated Chromium-Passivated Beryllium Powder (U)	23
VIII	Effect of Water-Treatment on Compatibility of Chromium-Coated Beryllium Powder with Ungelled Hydrazine (C)	25
IX	Effect of Water-Treatment on Compatibility of Beryllium Powder with Ungelled Hydrazine (C)	26
X	Beryllium Metal Content of Selected Water-Treated Chromium-Passivated Beryllium Samples (U)	26
XI	Compatibility of Hydrazine with Heat-Treated Beryllium Powders (U)	29
XII	Composition of Gaseous Reaction Products of Beryllium and Hydrazine (U)	31
XIII	Composition of Volatile Impurities of Beryllium (U)	32
XIV	Composition of Volatile Impurities on Beryllium (Brush FP-1755) (U)	34
XV	Gas Evolution Rate of Beryllium Powder GAC 4-88 in Ungelled Hydrazine (C)	36
XVI	Gas Evolution Rates of Beryllizine-21 (C)	36
XVII	Effect of Shearing on the Gas Evolution Rate of Beryllium in Gelled Hydrazine (C)	37
XVIII	Effect of Gelling Agents on the Gas Evolution Rates of Mixtures of Beryllium and Hydrazine (C)	39



# CONFIDENTIAL

Report AFRPL-TR-66-230

## LIST OF TABLES (cont.)

<u>Table</u>		<u>Page</u>
XIX	Dielectric Constant of Hydrazine after Contact with Beryllium Powders (C)	40
XX	Summary of Prototype Beryllizine-21 Batches (U)	43
XXI	Summary of Prototype Beryllizine-28 Batches (U)	44
XXII	Stability of Prototype Beryllizine-21 to Longitudinal Acceleration and Vibration (C)	47
XXIII	Gas Evolution Rates of Beryllizine-21 (77°F) (U)	48
XXIV	Chemical Stability Tests on One-Month-Old Prototype Beryllizine-21 (77°F) (U)	49
XXV	Bulk Growth Rates of Beryllizine-21 (77°F) (U)	50
XXVI	Chemical Stability Tests on Experimental Beryllizine-21 made with the Be Powders FP-555 and Cr-Coated R-4630 (C)	56
XXVII	Evaluation of Alternate Beryllium Powders in Beryllizine-21 (U)	58
XXVIII	Results of Chemical Analyses of Prototype Beryllizine (U)	62,63
XXIX	Test Data from Flow Loop Calibrations with Beryllizine (U)	81
XXX	Test Data from Flow Loop Testing in the Turbulent Regime (U)	85
XXXI	Hydraulic Performance, Modified Centrojet Injector (U)	90
XXXII	Hydraulic Performance, Modified Centrojet Injector (U)	91
XXXIII	Specific Impulse and Combustion Temperature as Functions of Beryllium Content in the $N_2O_4/N_2H_4$ + Be System (C)	94
XXXIV	Summary of Performance Calculations, $N_2O_4$ /Beryllizine-21 (U)	95
XXXV	Specific Impulse and Combustion Temperature as Functions of Beryllium Content in the 98% $H_2O_2/N_2H_4$ + Be System (C)	96
XXXVI	Summary of Performance Calculations 98% $H_2O_2$ /Beryllizine-28 (U)	99
XXXVII	$N_2O_4$ /Beryllizine-21 Test Conditions (U)	128
XXXVIII	$N_2O_4$ /Beryllizine-21 Test Results (U)	129
XXXIX	98% $H_2O_2$ /Beryllizine-28 Test Conditions and Results (U)	141

# CONFIDENTIAL

Report AFRPL-TR-66-230

## LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Chemical Stability Apparatus (U)	12
2	1/2-Gallon Laboratory Mixer (U)	46
3	Density of Prototype Beryllizine-21 (U)	51
4	Characteristic Flow Curves from Measurements with ASTM D-1092 Capillary Viscometer at 77°F (U)	52
5	Brookfield Limiting Flow Characteristic for Beryllizine-21 (77°F) (U)	54
6	Relationship of High Shear Viscosity to Yield Stress (77°F) (U)	55
7	Flow Loop (U)	65
8	Thixotropic Time Dependency (U)	68
9	Tube Wall "Slip Velocity" Effect (U)	69
10	Non-Newtonian Flow Diagram (U)	70
11	Adjusted Shearing Stress and Turbulent Velocity Relationship (U)	72
12	Correlation for Pseudoplastic Flow (U)	73
13	Adjusted Shearing Stress and Turbulent Velocity Relationship with Transition Region (U)	74
14	Flow Diameter Exponent in the Turbulent Region for Anomalous Fluids (U)	75
15	Alternative Correlation for Anomalous Fluids (U)	75
16	Method of Tube Flow Data Presentation (U)	76
17	Entrance Correction for Capillary Viscometer (U)	78
18	Beryllizine-21 Thixotropy (U)	79
19	Test Data, Water Flow (U)	82
20	Comparison of Flow Data (U)	83
21	Characteristic Flow Curve in the Turbulent Regime for Alumizine-43G and Beryllizine-21 (U)	86
22	Characteristic Flow Curve for Alumizine and Beryllizine-21 (U)	87
23	Comparison of Alumizine-43 $K_v$ with Water $K_v$ (U)	88
24	Theoretical Performance of $N_2O_4/0.79 N_2H_4 + 0.21 Be$ (C)	97

# CONFIDENTIAL

Report AFRPL-TR-66-230

## LIST OF FIGURES (cont.)

<u>Figure</u>		<u>Page</u>
25	Theoretical Performance of 98% $H_2O_2$ /0.72 $N_2H_4$ + 0.28 Be (C)	100
26	Modified Centrojet Injector (U)	106
27	Injector Assembly, Second Design (U)	107
28	Uncooled Thrust Chamber (U)	109
29	Test Stand and Thrust Chamber Assembly (U)	111
30	Test Stand and Thrust Chamber Assembly with 40:1 Area Ratio Nozzle (U)	112
31	Inserts, Thrust Chamber (U)	114
32	Inserts Assembled, Thrust Chamber (U)	115
33	Thrust Mount (U)	117
34	Vacuum Catch Tank and Gate Valve (U)	118
35	Exhaust Duct (U)	119
36	Test Stand Enclosure and Bellows (U)	120
37	Oxidizer Feed System (U)	121
38	Schematic, Oxidizer Feed System (U)	123
39	Fuel Feed System (U)	124
40	Schematic, Fuel Feed System (U)	125
41	Thrust Chamber Valves (U)	126
42	$N_2O_4$ /Beryllizine-21 Tests, Centrojet Injector (U)	130
43	$N_2O_4$ /Beryllizine-21 Tests, Second Injector (U)	131
44	Experimental Performance of $N_2O_4$ /Beryllizine-21 (U)	132
45	98% $H_2O_2$ /Beryllizine-28 Tests, Centrojet Injector (U)	142
46	Experimental Performance of 98% $H_2O_2$ /Beryllizine-28 (U)	143
47	Relationship of Combustion Temperature and Water Concentration with Mixture Ratio for the 98% $H_2O_2$ /Beryllizine-28 Propellant System (U)	153
48	Vacuum Specific Impulse versus Mixture Ratio as Predicted by Thermochemical, Ideal-Gas and Particle Flow Methods (U)	156

# CONFIDENTIAL

Report AFRPL-TR-66-230

## SECTION I

### INTRODUCTION

(C) In May 1965 the Aerojet-General Corporation commenced a 12-month program to (1) develop and characterize storable beryllium-containing fuels for use in advanced propulsion systems; (2) develop techniques for the ignition and combustion of the fuels with nitrogen tetroxide and 98% hydrogen peroxide; and (3) obtain data on the performance of the beryllium-containing fuels with these oxidizers at the 1000-lb thrust level.

(C) Two beryllium-containing fuels were to be developed during this program. Their beryllium concentrations were to be selected to give maximum theoretical specific impulse with  $N_2O_4$  and 98%  $H_2O_2$ , respectively, at a chamber pressure of 1000 psia.\* In each case the carrier was to be hydrazine, which was to be gelled with the most appropriate gelling agent available for this purpose.

(C) The program was divided into two major parts. One consisted of laboratory investigations directed toward propellant development. The other was concerned with the engineering investigations of ignition and combustion and of the flow characteristics of Beryllizine-21. A third part of the program was concerned with the industrial hygiene aspects of the handling of beryllium-containing propellant.

#### A. PROPELLANT DEVELOPMENT

(C) The objective of the propellant development part of this program was to bring Beryllizine-21 to the state of development of Alumizine-43, which is the aluminum-containing propellant developed by Aerojet prior to this program. The propellant development work was divided into the following tasks:

##### 1. Chemical Laboratory Investigations

a. (U) Survey and selection of propellant properties and characteristic criteria that should be incorporated into a realistic specification for Beryllizine.

b. (U) A survey of existing rocket propulsion systems to determine the range of acceleration and vibration conditions that may be experienced by a vehicle during transportation as well as flight.

(C) \*The optimum metal concentrations in the two fuels for use with  $N_2O_4$  and 98%  $H_2O_2$  are 21 and 28%, respectively (see section on theoretical performance calculations). Throughout this report these fuels are referred to as Beryllizine-21 and Beryllizine-28.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### I, A, Propellant Development (cont.)

c. (U) The preparation of a specification based on the criteria derived from the above surveys and considerations of other pertinent factors.

d. (U) Laboratory investigations required to formulate and characterize Beryllizine-21 and Beryllizine-28 to meet these criteria and to verify and further define the selection of propellant criteria.

#### 2. Propellant Mixing Technology

(U) The development of techniques and equipment for producing Beryllizine for the formulation and characterization investigations and for use in the engineering phases of the program.

#### 3. Secondary Propellant Development

(U) Formulation and characterization of Beryllizine-28 and the incorporation of pertinent data into the Beryllizine specification.

(U) All the objectives of the propellant development part of the program were not attained. When it was discovered, during the 8th program month, that the gelling agent selected for Beryllizine (Carbopol-940) was enhancing the gas evolution of the formulation, it became apparent that both the fuel development work and the ignition and combustion studies could not be completed within the program budget. During the 9th program month the Air Force project office requested Aerojet to consider the propellant development part of the program complete. This work was then terminated in favor of completion of the ignition and combustion investigations.

(U) The survey and selection of propellant properties and characteristic data, the survey of rocket propulsion systems, and the mixing technology investigations were completed; and a tentative Beryllizine specification was prepared in skeleton form (see Appendix C). It was intended that this specification would be completed during the course of the program, but this was not possible because the termination of the work occurred before the final fuel formulations were developed.

### B. ENGINEERING INVESTIGATIONS

(C) The principal objective of the engineering part of this program was to develop techniques for the ignition and combustion of Beryllizine with  $H_2O_4$  and 98%  $H_2O_2$ . Special as well as conventional techniques for the ignition of beryllium were to be investigated inasmuch as this may be the crux of the problem of efficient burning of the metal. A test program was to be undertaken to evaluate the optimum operating conditions and the most promising

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Report AFRPL-TR-66-230

## I, B, Engineering Investigations (cont.)

ignition and combustion techniques. When satisfactory performance was demonstrated, tests of a duration suitable to verify injector performance and durability were to be conducted. Although the steady-state performance was to be evaluated, emphasis was to be placed on the problem of ignition and combustion of Beryllizine.

(U) A second objective of the engineering part of the program was to establish the flow characteristics of Beryllizine-21. The rheological properties of the fuel were to be measured with laboratory equipment to predict the flow behavior. Tests were then to be conducted in a flow apparatus for verification of the flow behavior in standard plumbing configurations. In addition, the flow behavior of Beryllizine-21 was to be evaluated in injector elements and manifolds to determine pressure drop and the problems of erosion, clogging, separation, etc. A survey of the state of the art in flow-measurement methods for gelled metalized fuels was to be made to determine the most reliable and practical method available.

## C. INDUSTRIAL HYGIENE

(U) The objective of the industrial hygiene part of the program was to assess the difficulties and hazards associated with the handling of toxic propellants and their components. Equipment design and operating procedures were to be established taking these factors into account, and their safety was to be verified during the course of the program to the extent that this was feasible.

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Report AFRPL-TR-66-230

## SECTION II

### SUMMARY

#### A. CHEMICAL LABORATORY INVESTIGATIONS

(U) The purpose of the propellant phase of this program was (1) to provide prototype Beryllizine fuels that were suitable for use in the engineering investigations, and (2) to develop and characterize chemically and mechanically stable beryllium-hydrazine fuels.

(C) The prototype Beryllizine fuels were prepared and characterized early in the program in order that the engineering investigations could be performed simultaneously with the propellant development program. Commercial anhydrous hydrazine meeting the requirements of MIL-P-27412 (USAF), Brush FP-1755 beryllium powder, and Carbopol-940 gelling agent were used. This combination of components, along with the processing procedure developed under previous programs, represented the best state-of-the-art materials and methods. Characterization of the prototype fuels showed that they were suitable for the engineering investigations but that storage had to be restricted to 2 months because of excessive gas evolution and bulk growth.

(C) Concurrent with the program to supply prototype Beryllizine for the engineering investigations, a research program was conducted to investigate the causes of incompatibility of the propellant components and to develop methods for improving the chemical stability. The first part of the program was an investigation of the sources of gas evolved by mixtures of beryllium and ungelled hydrazine and of materials and techniques for improving the chemical compatibility of these materials. The objective was to utilize the results of these studies to replace the prototype Beryllizine formulations by advanced formulations that would meet all of the program goals, including gas evolution and bulk growth rates. The values selected for the goals were  $1.5 \times 10^{-4}$  cc/lb-min for the gas evolution rate and  $5 \times 10^{-6}$  %/min for the bulk growth rate, both at 77°F. These were based on criteria for Alumizine-43G meeting the requirement of military specification MIL-P-27412 (USAF).

(C) The chemical compatibility investigations were conducted by studying the reaction of ungelled hydrazine with (1) untreated beryllium powders, (2) chemically treated beryllium powders, (3) coated beryllium powders, and (4) heat-treated beryllium powders. In the investigation of the compatibility of ungelled hydrazine with untreated beryllium powder it was found that, in general, the purest beryllium powders are the most compatible, and that magnesium and beryllium carbide appear to be most responsible for high gas evolution rates. Treatment of beryllium with anhydrous hydrazine, 5% nitric acid, or 30% hydrogen peroxide was ineffective or increased the reactivity of the beryllium powder with hydrazine. Of the chemical treatments of beryllium powder, only the water extraction and alcoholic KOH treatments were effective. The water treatment of chromium-coated beryllium powders,

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Report AFRPL-TR-66-230

### II, A, Chemical Laboratory Investigations (cont.)

particularly those with low contents of impurities, was the most successful of the methods investigated. By proper selection of the beryllium powder and proper water treatment the average gas evolution rate of beryllium-hydrazine mixtures was reduced to  $1.5 \times 10^{-4}$  cc/lb-min at 90°F. The alcoholic potassium hydroxide treatment also improved the compatibility of beryllium-hydrazine mixtures, but not as much as the water treatment. Neither the organic poly-semicarbazide coating nor the metallic aluminum coating was effective in reducing gas evolution rates, and investigations of the heat-treatment of beryllium powders were inconclusive.

(C) When Carbopol-940 was used to gel mixtures of beryllium in hydrazine it became apparent that the Carbopol adversely affected the chemical stability. The substitution of CPAA-12 and CP\* for the Carbopol corrected this problem.

### B. ENGINEERING INVESTIGATIONS

(C) The objectives of the engineering investigations were to characterize the flow behavior of Beryllizine-21 and to develop techniques for the ignition and efficient combustion of the  $N_2O_4$ /Beryllizine-21 and 98%  $H_2O_2$ /Beryllizine-28 systems.

(C) Flow testing was conducted with Beryllizine-21 in the laminar and turbulent regions. The ASTM D-1092 capillary viscometer was used to characterize flow in the laminar region. Alumizine-43G was also flowed for comparison purposes and the results show that these fluids have similar properties in the laminar regime.

(C) A flow loop built for flow testing in the turbulent region consisted of a fuel feed system, a test section of 1/4-in.-dia stainless steel tubing, and a catch tank. The loop was first calibrated with Beryllizine-21 in the laminar region and the results compared with data measured with the capillary viscometer. The flow loop and capillary data were consistent, indicating the flow loop was operating properly. Flow loop tests were then conducted with Beryllizine-21 in the turbulent regime. The results were compared with available turbulent data for Alumizine-43G and the flow properties were found to be nearly the same for fuels having comparable yield stress values. Furthermore, the points of transition from laminar to turbulent flow were found to occur at nearly identical shear rates.

\*CPAA-12 and CP are derivatives of cross-linked polyacrylic acid prepared by the Aerojet-General Corporation. Vander Wall, E. M., Lucas, J. M., and Fish, W. R., "Improved Gelling Agents for Hydrazine and Hydrazine-Type Fuels," preprints of the 8th Liquid Propulsion Symposium, CPIA Publication No. 121, Vol. I, p. 431, October 1966 (paper Unclassified, preprint volume Confidential).



## CONFIDENTIAL

Report AFRPL-TR-66-230

### II, B, Engineering Investigations (cont.)

(C) The ignition and combustion investigations were commenced with evaluations of the theoretical data available for the propellant systems. The computer program which calculates chemical compositions and performance parameters was then updated with the new thermodynamic data. The updated program was used to optimize the formulations for the fuels to be used with  $N_2O_4$  and 98%  $H_2O_2$  as oxidizers at 1000-psia chamber pressure. Beryllizine containing 21% of beryllium powder was selected for use with  $N_2O_4$  and Beryllizine containing 28% of the metal was selected for use with 98%  $H_2O_2$ . Values of theoretical performance were calculated for chamber pressures of 500, 1000, and 1500 psia over a mixture ratio range of 0.5 to 1.25. These data were used for engineering design purposes and in evaluations of experimental results.

(C) It is generally accepted that beryllium particles are coated with beryllium oxide, which must melt before ignition of the beryllium metal can occur. Unfortunately, the reaction temperatures of the liquid components of the  $N_2O_4$ /Beryllizine-21 and the 98%  $H_2O_2$ /Beryllizine-28 propellant systems do not approach the melting point of the beryllium oxide at mixture ratios attractive for thrust chamber operation. Early in the program many methods were considered as solutions to this problem. They included modification of the beryllium particles, such as chromium or aluminum coatings; modification of the fuel formulations, such as the addition of a small percentage of aluminum powder; and tailoring part of the injector geometry to produce conditions favorable for ignition of the metal. After a thorough study of these methods, the approach of tailoring part of the injector geometry to promote ignition was chosen as the simplest and most practical method available. The section of the injector which produces the conditions favorable for ignition of the beryllium particles is known as the hot-core.

(C) Two injectors were built for use in the test program. The first was a modification of the Centrojete injector developed by U. S. NOTS, China Lake, California. The second injector was patterned after an injector concept developed by Aerojet for use with the  $N_2O_4$ /Alumizine-43G propellant system. The faces of both injectors were protected by transpiration cooling with oxidizer, and both designs contained a hot-core located in the central portion of the injector face. The injectors were tested in conjunction with an uncooled thrust chamber assembly and a nozzle extension which produced an area ratio of 40:1. The thrust chamber assembly was designed to produce 1000 lb of thrust at 1000 psia chamber pressure.

(C) Twenty-five tests were conducted with  $N_2O_4$ /Beryllizine-21 using the two injectors and the uncooled thrust chamber. The tests were conducted at mixture ratios which averaged approximately 0.6 and the test durations were approximately 2 sec. The hot-cores of both injectors produced conditions which ignited the beryllium metal, but the Centrojete delivered the highest percentage of theoretical performance. The percentage of theoretical

## CONFIDENTIAL

Report AFRPL-TR-66-230

### II, B, Engineering Investigations (cont.)

specific impulse increased with increasing chamber pressure. The Centrojet delivered 86.3% of theoretical performance, which corresponds to 321 sec of delivered specific impulse.

(C) One test was performed with the  $N_2O_4$ /Alumizine-43G system for calibration purposes. Performance efficiencies of 92 to 93% have been consistently demonstrated at higher levels of thrust with  $N_2O_4$ /Alumizine-43G. It had been calculated that the performance efficiency delivered by the  $N_2O_4$ /Beryllizine-21 propellant system was acceptable because the theoretical performance had been reduced by approximately 6% due to solid-particle flow through the small nozzle. On the other hand, it had been predicted that a performance efficiency on the order of 85% was all that could be expected from the  $N_2O_4$ /Alumizine-43G system using the 1000-lb thrust chamber because of flow losses. The test resulted in 85.3% of theoretical performance, which gives confidence in the results of the tests with  $N_2O_4$ /Beryllizine-21.

(C) Eleven tests were conducted with the 98%  $H_2O_2$ /Beryllizine-28 propellant system; however, only five of these tests resulted in valid test data. This series of tests consisted of nine thrust chamber tests and two tests of the hot-core alone.

(C) The initial three of the nine thrust chamber tests did not produce valid data because of hard starts which damaged the injector face, or improper operation of other equipment which prematurely terminated the test. Preliminary to the start transient of the fourth test, a detonation occurred in the combustion chamber which severely damaged the test equipment. Subsequent to the explosion, the equipment was rebuilt and a method of preventing hard starts with Alclo igniters was adopted. After a group of three igniter tests to develop a reliable ignition sensor, the first thrust chamber test was successfully conducted.

(U) During a routine inspection of the test equipment preliminary to the fifth test, fatigue cracks were found around the periphery of the oxidizer tank. A new tank was installed and prepared for testing. During the preparatory activities for the fifth test, the oxidizer tank exploded and severely damaged the oxidizer feed system. Enough components remained undamaged to reconstruct the system and resume testing. The remaining four ignition and combustion tests using the complete thrust chamber assembly were conducted without incident and produced valid data.

(C) Even though the third thrust chamber test (1K-2B-129) was terminated prematurely by a safety shutdown device, beryllium oxide was found inside the combustion chamber and vacuum enclosure. The presence of oxide indicates ignition of the beryllium powder prior to shutdown and proper operation of the hot-core with 98%  $H_2O_2$  and Beryllizine-28.

# CONFIDENTIAL

Report AFRPL-TR-66-230

## II, B, Engineering Investigations (cont.)

(C) The tests were conducted over a mixture ratio range of 0.65 to 1.1. The highest performance efficiency (90.3%) was obtained at high mixture ratio and the lowest efficiency (82.8%) was obtained at low ratio. A test with the combustion chamber lengthened from 12.5 in. to 22.5 in. was conducted at a mixture ratio of 0.66 and resulted in a performance efficiency of 86.8%. The other tests were conducted at different ratios within the range to study the effect of mixture ratio on performance efficiency. A definite trend was established and it shows that performance efficiency rises with increasing mixture ratio. It is felt that combustion is improved by the gross increases of water vapor concentration in the combustion products rather than the slight increases in combustion temperature which are obtained when proceeding from low to high mixture ratio.

# CONFIDENTIAL

Report AFRPL-TR-66-230

## SECTION III

### TECHNICAL DISCUSSION

#### A. CHEMICAL LABORATORY INVESTIGATIONS

##### 1. Objectives

(U) The objectives of the chemical laboratory investigations were to (1) develop and characterize Beryllizine-21 and -28 that will satisfy the operational conditions of advanced propulsion systems, (2) prepare a specification for the Beryllizine fuels, and (3) develop a mixing procedure and equipment for producing the propellant required for the engineering phase of this program.

##### 2. Criteria Survey and Preparation of Specification

(C) A survey of existing and proposed propulsion and missile systems was conducted to define the operational and environmental conditions that the Beryllizine propellant must endure. On the basis of this study, pertinent criteria for propellant properties and characteristics were selected and incorporated into a Beryllizine Fuel Criteria Document, which is included in this report as Appendix A. This document is a working record of the approved criteria and limiting values which was revised as required during the course of the program. The propellant criteria are divided into three categories: (1) propellant characteristics, such as mechanical stability, chemical stability, and rheological behavior; (2) propellant composition, including component concentration and quality restrictions; and (3) propellant properties, including density, yield stress, and other properties essential to the characterization of the propellant for quality assurance purposes. The criteria document also includes a section on test instruments required for measuring the properties of the propellants. The values of  $1.5 \times 10^{-4}$  cc/lb-min for the gas evolution rate and  $5 \times 10^{-6}$  %/min for bulk growth rate are based on similar criteria for Alumizine meeting Specification MIL-P-27412 (USAF). The requirement that the propellant be stable for a minimum of one year is also based on Alumizine. The minimum yield stress limit, 1400 dynes/cm<sup>2</sup>, is established to ensure that the propellant will be mechanically stable and the maximum yield stress value, 2000 dynes/cm<sup>2</sup>, is established to ensure that the propellant will flow properly through pumps, lines and orifices. A second requirement to ensure proper flow is that the high shear viscosity falls within specified minimum and maximum values as measured by the Ferranti-Shirley viscometer and by the ASTM D-1092 capillary viscometer. Values for both viscometers are included so that either instrument may be used.

(C) Using the propellant criteria and associated limiting values, a tentative military specification for Beryllizine fuel was prepared in the format of the Alumizine specification, MIL-P-27412 (USAF). The purpose of this specification, which is included as Appendix C, is to specify the essential property, composition, and test requirements necessary to define Beryllizine

CONFIDENTIAL

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

for developmental purposes. This specification was prepared in skeleton form at the beginning of the program. It was intended that it would be completed during the course of the program, but this was not possible because the propellant part of the program was considered to have been completed before the final fuel formulations were developed. The limits on the concentrations of components and on the physical properties listed in Table I of the specification are based on the results obtained when various concentrations of gelling agent were used in making prototype Beryllizine. The physical properties are based on the allowable component concentrations, preliminary testing of mechanical stability, and flow behavior of the prototype Beryllizine. The upper limit on yield stress, 2000 dynes/cm<sup>2</sup>, is a control on excessive gel structure and corresponds to the limit specified for Alumizine-43G in MIL-P-27412 (USAF).

(C) In order to determine appropriate vibration and acceleration criteria for use in the preparation of the Beryllizine Fuel Criteria Document a study of existing missile systems was initiated. This study was a survey of actual vibration and acceleration conditions experienced by vehicle tankage both in flight and during transportation. These data were to be used to establish criteria for tests that would simulate the vibration and acceleration conditions experienced in vehicle service. These tests could then be used to assess the stability of the metalized fuel. This part of the study was not completed.

(C) A summary of the information and a brief discussion of the data are contained in Appendix B. The principal conclusions were that the maximum longitudinal acceleration experienced by liquid missile systems is 12 g, and the vibrations encountered by missiles in transportation and during flight are almost entirely random. They have the following limiting characteristics:

	<u>Frequency Bands, cps</u>	
	<u>5-500</u>	<u>500-2000</u>
Acceleration Spectral Density, g <sup>2</sup> /cps	0.5	1.25
Maximum Peak Acceleration, g rms	5	40

### 3. Propellant Formulation and Characterization

#### a. Compatibility of Propellant Components

(C) Previous investigations of Beryllizine indicated that there are two principal problems in the preparation of storable formulations gelled with Carbopol-940. One is related to the adverse effects of electrolytes on the gelling capacity of Carbopol-940. The other is the reaction of hydrazine with beryllium or impurities in the beryllium which yield gaseous decomposition products. During the course of this investigation it became apparent that the gelling agent, Carbopol-940, was also involved in a gas-producing reaction in the presence of beryllium and hydrazine.

CONFIDENTIAL

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

(C) Five approaches were taken to solve the problem of gas evolution by beryllium/hydrazine gels as follows: (1) selection of those beryllium powders which have least reactivity with hydrazine; (2) chemical treatment of the beryllium powders either to remove the impurities responsible for the decomposition of the hydrazine or to change the surface that is responsible for the reactivity; (3) heating of the powders to reduce the reactivity; (4) elimination or reduction of the contact of the beryllium with hydrazine; and (5) modification of the gelling agent to reduce or eliminate its reactivity with beryllium/hydrazine mixtures. Investigations using these approaches to reduce gas evolution are discussed in the following paragraphs.

#### (1) Beryllium Powder-Hydrazine Compatibility Investigations

(C) The purposes of this phase of the investigation were to (1) establish the relative reactivities of selected beryllium powders with hydrazine and provide baseline data with which to compare data for treated powders, (2) determine the value of various treatments in reducing the incompatibility problem, (3) determine the value of various coatings on the powders, and (4) determine the effects of various impurities present in the beryllium powders.

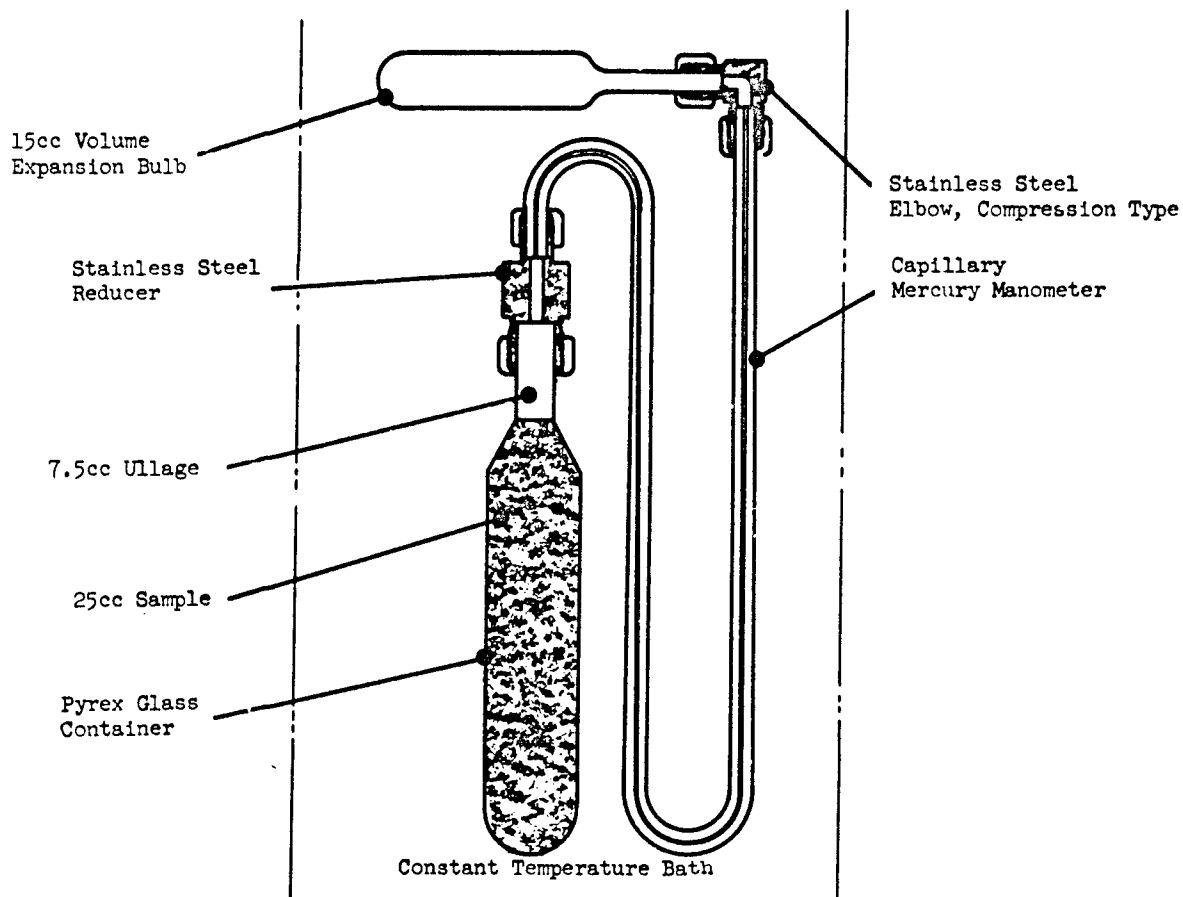
(U) The apparatus used for the determination of chemical compatibility is illustrated in Figure 1. Prior to use, the pyrex sample container was passivated with a 10% aqueous solution of hydrazine, rinsed with distilled water and methanol and dried in an oven. A weighed sample of metal powder was then placed in the container and evacuated for 1 hr at ambient temperature. After the vacuum was relieved by the introduction of nitrogen gas, the container was transferred to a nitrogen filled glove bag where hydrazine was added and the apparatus was assembled. The apparatus was then placed in a constant temperature bath at 90°F and allowed to equilibrate for at least 2 hr before the initial height of the mercury in the manometer was measured. Subsequent pressure measurements were made twice a week.

(U) Unless otherwise specified, the beryllium concentration was 28% by weight, the sample size was 25 ml and the ullage was 23% of the total volume of the sample container.

(U) Changes in pressure in the compatibility apparatus were determined by measuring the height of the mercury column with a cathetometer and recorded in millimeters of mercury. These data were plotted graphically with respect to time. To obtain the gas evolution rates, presented as cc/lb-min, pressure changes in millimeters of mercury were converted to volumes of gas at 77°F and one atmosphere (nTP) and computed per pound of sample per minute during the 24-hour period preceding the day for which the data are reported.

**CONFIDENTIAL**

Report AFRPL-TR-66-230



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Figure 1. Chemical Stability Apparatus

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## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

#### (a) Compatibility of Untreated Beryllium Powders with Ungelled Hydrazine

(C) Initially eight samples of commercially available beryllium powders were chosen to investigate their chemical compatibility with hydrazine. They represented variations in the contents of major impurities and surface treatment by the manufacturer. The powders selected were the Brush Beryllium Company FP-1755 used in the prototype formulation, The General Astrometals Corporation (GAC) Grade 4 powder from Lot 88, and six types of Beryllium Corporation of America (Berylco) powder, Grade PS-97 or PS-98. Two of these were oxide-coated, two were chromium-coated, one was treated with a low-boiling amine by Berylco and the other was both chromium-coated and amine-treated. The manufacturers' analyses for these powders are presented in Table I.

(C) The GAC 4-88 powder, the purest powder used in these experiments, was the most compatible with hydrazine. The gas evolution rate was in the range from  $3.6$  to  $7.5 \times 10^{-4}$  cc/lb-min for the 60-day test period. In the case of the Brush FP-1755 the initial rates were of the same magnitude, but they increased during 3-5 week periods to the range from  $19$  to  $42 \times 10^{-4}$  cc/lb-min. The oxide-coated Berylco powders, R-2039 and R-3951, evolved gas at such high rates that the experiments were terminated after 4-8 days. The rates for the amine-treated material, R-4297, were extremely high. The rates for the amine-treated chromium-coated powder, R-4298, were almost as low as those for the GAC powder, but this was attributed to factors other than the treatment with amine. In the case of the chromium-coated Berylco powders, R-3951 and R-3892, the compatibility appeared to be related not only to the presence of the coating, but also to the magnesium content. The powder with the lower magnesium content, R-3951, had a gas evolution rate in the range from  $11$  to  $13 \times 10^{-4}$  cc/lb-min (comparable to the rate for the chromium-coated, amine-treated powder, which also had a relatively low magnesium content), whereas the powder with the higher magnesium content had a rate twice as high. The gas evolution data for these samples are included in Table II.

(C) Subsequent to the experiments just described, eight additional powder samples were examined for their compatibility with hydrazine. These were all chromium-coated Berylco powders, Grade PS-98, which were tested to further examine the relation of gas evolution rate to the level of impurities and to the passivation treatments which are discussed below. It was not possible in this work to establish unequivocally the relation of magnesium content of beryllium powders to compatibility, but only that in the case of beryllium manufactured by the magnesium reduction method, the lowest gas evolution rates were observed with the chromium-coated powders having low contents of impurities. The impurities that were most suspect as sources of evolved gas in hydrazine were magnesium and carbon.



**CONFIDENTIAL**

Report AFRPL-TR-66-230

TABLE I

COMPOSITION OF SELECTED BERYLLIUM POWDERS (u)

Sample*	Composition, wt. %							Particle Size, Microns, ave	Remarks
	BeO	C	Fe	Al	Mg	Ni	Mn		
4.88 <sup>a</sup>	1.74	0.015	0.033	0.049	0.001	0.027	0.001	0.016	20
R-3359 <sup>b</sup>	1.81	0.47	0.048	0.068	0.095	0.004	0.008	0.005	9.4
R-2039 <sup>b</sup>	0.65	0.65	0.076	0.079	0.8	0.007	0.010	0.005	9.5
R-3951 <sup>b</sup>	0.5	0.25	0.110	0.051	0.037	0.021	0.16	0.290	0.094
R-3892 <sup>b</sup>	0.5	0.25	0.062	0.028	0.250	0.009	0.009	0.290	0.025
FP-1755 <sup>c</sup>	1.35	0.11	0.14	0.07	0.01	---	---	0.07	17
R-4297 <sup>b</sup>	1.34	0.38	0.093	0.073	0.190	0.015	0.013	0.006	0.053
R-4298 <sup>b</sup>	0.5	0.25	0.070	0.043	0.095	0.020	0.015	0.290	0.055
R-2045 <sup>b</sup>	0.5	0.50	0.130	0.053	0.036	0.019	0.011	0.385	0.045
R-2407 <sup>b</sup>	0.5	0.54	0.115	0.082	0.046	0.011	0.012	0.440	0.047
R-2508 <sup>b</sup>	0.5	0.19	0.137	0.088	0.005	0.015	0.010	0.150	0.028
R-2001 <sup>b</sup>	0.5	0.84	0.225	0.036	0.9	0.016	0.008	0.420	0.025
R-4460 <sup>b</sup>	0.5	0.24	0.102	0.129	0.016	0.010	0.013	0.285	0.035
R-4630 <sup>b</sup>	0.5	0.20	0.049	0.070	0.034	0.010	0.018	0.300	0.090
R-4635 <sup>b</sup>	0.5	0.21	0.052	0.071	0.025	0.010	0.013	0.270	0.041
R-4639 <sup>b</sup>	0.5	0.21	0.065	0.072	0.034	0.010	0.010	0.290	0.058

\*Sources: a--General Astrometals Corporation  
b--Beryllium Corporation  
c--Brush Beryllium Company

**CONFIDENTIAL**

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE II

## COMPATIBILITY OF HYDRAZINE WITH UNTREATED BERYLLIUM POWDERS (C)

Test Conditions: Pyrex Container; 90°F; 25cc Sample; 28% Be, 72%  $N_2H_4$ ; 23% Ullage

Sample	Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>														
	Time, days														
	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60
FP-1755/BBC	5.4	8.6	7.5	12.9	18.3	19.4	*								
FP-1755/BBC	8.6	6.4	9.7	18.3	12.0	29.0	38.7	42	*						
FP-1755/BBC	5.4	9.7	12.9	17.2	17.2	19.4	26.9	32.3	*						
R-3359/Berylco	32.3	25.8	*												
R-2039/Berylco	49.5	*													
R-2039/Berylco	64.6	*													
R-3951/Berylco	10.8	12.9	12.9	12.9	11.8	11.8	*								
R-3951/Berylco	9.1	9.1	8.6	9.5	9.5	9.5	7.6	7.6	8.6	8.6	8.6	8.6	6.4	6.4	*
R-3892/Berylco	24.7	25.8	*												
GAC 4-88	6.4	6.4	6.4	7.5	6.4	6.4	5.5	5.5	5.5	5.4	3.9	3.9	3.9	3.9	3.6
GAC 4-88	5.4	7.5	6.4	6.4	8.6	6.4	6.4	7.5	*						
R-4297/Berylco	Pressure rise too rapid to warrant calculation														
R-4297/Berylco	Pressure rise too rapid to warrant calculation														
R-4298/Berylco	9.7	11.8	8.6	8.6	*										
R-2045/Berylco	22.9	22.9	22.9	22.9	22.9	*									
R-2C01/Berylco	58	*													
R-2508/Berylco	8.4	8.4	8.4	14.2	14.2	*									
R-2508/Berylco	7.0	7.0	7.0	11.0	11.0	11.0	15	15	15	2.5	5.8	5.8	5.8	5.8	*
R-2407/Berylco	8.7	8.7	8.7	8.7	8.7	8.7	8.7	*							
R-2407/Berylco	12	12	12	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7	*
R-4460/Berylco	18	18	18	*											
R-4460/Berylco	16	16	16	*											
R-4630/Berylco	8.4	6.3	7.3												
R-4630/Berylco	7.3	8.4	8.4												
R-4635/Berylco	19	*													
R-4635/Berylco	27	19	*												
R-4639/Berylco	12	15	15												
R-4639/Berylco	8.4	15	18												

\* Discontinued

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

### (b) Effect of Chemical Treatment of Beryllium Powder on Compatibility with Hydrazine

(C) Because of the relatively high gas evolution rates observed for the samples of untreated beryllium powder, a series of tests was conducted in which these powders were leached to remove reaction impurities or to passivate active sites prior to compatibility testing in hydrazine. The materials used for this purpose were (1) dilute nitric acid, (2) dilute hydrogen peroxide, (3) hydrazine, (4) alcoholic potassium hydroxide, and (5) water.

#### 1 Effect of Dilute Nitric Acid

(C) Three samples of beryllium powder were leached with 5% aqueous nitric acid: FP-1755, which had a low initial gas evolution rate in the untreated condition and was not chromium-passivated; R-2039, which had a high gas evolution rate in the untreated condition and was not chromium passivated; and R-3951, a chromium-passivated powder that had a low gas evolution rate in the untreated condition. A 10-gm quantity of each of the powders was allowed to stand about 16 hr in 20 ml of dilute nitric acid at 90°F. After this step was repeated twice more, the powders were rinsed three times with distilled water and dried in a vacuum oven at 100°C. The samples were then stored in a nitrogen atmosphere until used. The tests of their compatibility with hydrazine were conducted in the manner described for the untreated powder.

(U) The results presented in Table III, along with comparable data for the untreated powders, show that the treatment with acid caused the powders to become significantly more reactive with hydrazine. These tests were terminated after four days, because of the excessively high pressures developed, and no further testing with the dilute nitric acid treatment was conducted.

TABLE III

COMPATIBILITY OF HYDRAZINE WITH BERYLLIUM  
POWDERS TREATED WITH 5% NITRIC ACID (C)

Beryllium Sample Source	Gas Evolution Rate, cc/lb/min x 10 <sup>4</sup>	
	4th Day Data	
	Treated Material	Untreated Material
FP-1755/BBC	68.9	8.6
R-2039/Berylco	60.2	49.5
R-3951/Berylco	31.2	10.8

(CONFIDENTIAL TABLE)

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

#### 2 Effect of Dilute Hydrogen Peroxide

(C) The effect of an oxidizing agent was investigated by treating three of the selected beryllium powders with 30% hydrogen peroxide in the manner described for the nitric acid treatment. Only oxide coated samples were used in these tests. When the treated powders were tested for compatibility in hydrazine, two samples had gas evolution rates higher than those for the untreated powders, whereas the gas evolution rate was decreased in the case of the third powder to about one-third that for the same powder in the untreated condition (see Table IV). In view of these results, it was concluded that further work with the peroxide treatment was not warranted.

TABLE IV

#### COMPATIBILITY OF HYDRAZINE WITH BERYLLIUM POWDERS TREATED WITH 30% HYDROGEN PEROXIDE (C)

Test Conditions: Pyrex Container; 90°F; 25 cc sample; 28% be, 72%  $N_2H_4$ ;  
23% ullage

Beryllium Sample Source	Gas Evolution Rate, cc/lb/min x $10^4$			Untreated Be 4th Day Data
	Time, Days <u>4</u>	<u>8</u>	<u>12</u>	
FP-1755	19.4	19.4	26.6*	8.6
R-3359	66.7*			32.3
R-2039	18.3*			49.5
Berylco				

\*Discontinued

(CONFIDENTIAL TABLE)

#### 3 Effect of Hydrazine

(C) Investigations prior to this program had shown that the dielectric constant of hydrazine is increased when beryllium powders are leached with this fuel. Although recent experiments have not confirmed this observation, it was believed that the ionic impurities causing the change in the dielectric constant of the hydrazine might be responsible, at least in part, for the incompatibility with beryllium. Six of the selected beryllium powders were treated four times with hydrazine in the manner described for the nitric acid and peroxide treatments, and tested for compatibility with hydrazine.

CONFIDENTIAL

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

TABLE V

### COMPATIBILITY OF HYDRAZINE WITH BERYLLIUM POWDERS TREATED WITH HYDRAZINE (C)

Test Conditions: Pyrex Container; 90°F; 25 cc sample; 28% Be, 72% N<sub>2</sub>H<sub>4</sub>;  
23% ullage

Beryllium Sample Source	Gas Evolution Rate, cc/lb/min x 10 <sup>4</sup>						Untreated Be 4th Day Data
	Time (Days)						
	4	8	12	16	20	24	
FP-1755/BBC	32.3	35.5	*				8.6
R-3359/Berylco	51.6	*					32.3
R-2039/Berylco	35.5	*					49.5
R-3951/Berylco	7.5	8.1	10.7	9.6	9.6	8.9	10.8
R-3892/Berylco	17.2	16.1	16.1	12.9	*		24.7
GAC 4-88/GAC	22.6	23.5	22.4	*			6.4

\*Discontinued

(CONFIDENTIAL TABLE)

(C) The results of these experiments, which are presented in Table V, are anomalous, particularly with respect to samples GAC 4-88 and R-3359, which showed significantly higher gas evolution rates following the treatment with hydrazine. It is obvious, however, that the use of hydrazine as a leaching agent has little or no beneficial effect on the compatibility of beryllium powder with hydrazine.

#### 4 Effect of Alcoholic Potassium Hydroxide Treatment

(C) Of the five materials used to treat beryllium powder for the purpose of improving its compatibility with hydrazine, only the alcoholic potassium hydroxide treatment and the water treatment were effective. The water treatment is discussed in the next section.

(C) Two types of caustic treatment were used. In the first treatment a 10-gm quantity of each of the powders was allowed to stand about 16 hr in 20 ml of alcoholic 5% potassium hydroxide at 90°F. This step was repeated three times. The powders were then rinsed three times with ethanol and, in some cases, with water and dried in a vacuum oven at 100°C. The samples were then stored in a nitrogen atmosphere until used. The second treatment was to add 10 gm of the selected beryllium powder to 60 ml of alcoholic 5% KOH and reflux the mixture for 16 hr (173°F). In some cases

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

the treated beryllium powder was then leached with alcohol using a Soxhlet extraction apparatus. Other variations of the rinsing procedure are noted on Table VI, along with the results of the compatibility tests.

(C) Samples R-3359, R-2039, GAC 4-88, and FP-1755 are beryllium powder samples which have not been passivated by the vendor and can be considered oxide-coated. The results obtained with these powders showed no substantial improvement in compatibility after caustic treatment. It was concluded that this treatment was not effective with the oxide-coated beryllium powders. Therefore, work on the caustic-treated oxide-coated beryllium powders was discontinued.

(C) The results of work with the untreated powder showed that chromium passivation of beryllium powders was fairly effective in reducing the reactivity of the powders with hydrazine if the magnesium and carbon contents of the beryllium were low, and that when these chromium-passivated powders were treated with alcoholic potassium hydroxide further improvement in compatibility with hydrazine could be expected. Also, analyses of the metallic beryllium content of caustic-treated beryllium powders showed that the loss of beryllium metal was not excessively high. In view of these encouraging findings, further work was initiated to determine the effects of varying the temperature of the caustic treatment and the techniques for removing the caustic residues from the beryllium powder.

(C) The samples selected for this work were R-3951 and R-4460. Sample R-3951 was selected because it was the most compatible chromium-passivated beryllium powder after caustic treatment. R-4460 was selected because it represented the best chromium-passivated beryllium powder available in significant quantities. Two pounds of this powder, enough to prepare a batch of gel, were purchased. The carbon and magnesium contents were specified not to exceed 0.25 and 0.037%, respectively. The other impurities were specified not to exceed the levels of impurities presented in Beryllium Corporation of America material R-3951 (see Table I for the analysis of this material). R-4460 met these requirements except for the aluminum content.

(C) The results of tests with these powders indicate that the best compatibility with hydrazine is achieved by treating the chromium-passivated beryllium powder with refluxing alcoholic 5% KOH, and then rinsing or leaching the powder with water (Table VII). Regardless of the type of treatment used to remove the caustic residues, the results show that thorough removal is essential if good compatibility with hydrazine is to be achieved. As a result of this work it appears that a gas evolution rate of  $4 \times 10^{-4}$  to  $6 \times 10^{-4}$  cc/lb-min can be achieved using a chromium-passivated beryllium powder of low magnesium and carbon contents, if it is treated with alcoholic 5% potassium hydroxide and then thoroughly washed with water.

CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE VI  
COMPATIBILITY OF HYDRAZINE WITH BERYLLIUM POWDERS TREATED  
WITH ALCOHOLIC POTASSIUM HYDROXIDE (C)

Test Conditions: Pyrex container; 90°F; 25cc sample; 28% Be, 72% H<sub>2</sub>O; 235 Ullage

Sample	Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>											
	8	12	16	20	24	28	32	36	40	44	48	52
R-3559, Berylico	98.3	56.2	42.1	31.6	31.6	31.6	31.6	•				
R-3359, Berylico	23.7	38.7	•									
R-3892, Berylico	4.1	4.1	0.4	5.5	5.5	5.5	7.2	5.5	5.5	5.5	5.5	5.5
R-3892, Berylico	2.2	2.2	2.2	2.7	2.7	2.7	2.7	2.7	2.7	2.7	2.7	2.7
4-88, GAC	5.4	4.3	4.3	4.3	3.2	3.2	4.3	4.3	5.2	•		through 93rd day*
4-88, GAC	1.1	2.2	4.4	4.4	3.8	3.8	3.8	3.8	3.8	3.8	3.8	3.8
PF-1755, NBC	5.4	5.4	5.4	4.3	4.3	4.3	4.7	12.1	26.9	•		
PF-1755, NBC	2.1	2.1	2.1	3.7	20.4	20.4	•					
R-2039, Berylico	7.5	8.6	11.0	11.8	•							
R-2039, Berylico	31.2	•										
R-2039, Berylico	11.8	16.0	16.0	20.4	•	Refluxed at 173°F						
R-2039, Berylico	56.0	•	Refluxed with alcohol only									
R-3951, Berylico	2.4	4.3	2.1	2.1	3.2	1.6	1.6	2.1	1.9	0.8	•	
R-3951, Berylico	1.1	2.2	1.2	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
R-3951, Berylico	5.2	5.2	5.2	5.2	6.8	6.8	6.8	6.8	6.3	Refluxed at 173°F		through 93rd day*
R-3951, Berylico	12	12	12	9.3	9.3	•	Leached with alcohol					
R-3951, Berylico	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
												• Refluxed at 173°F and then leached with alcohol.
R-3951, Berylico	2.1	4.2	4.7	4.7	4.7	4.7	4.7	4.7	4.7	4.7	4.7	4.7
												• Aqueous KOH followed by a H <sub>2</sub> O rinse.
R-3951, Berylico	1.0	1.0	2.1	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
												• Refluxed at 173°F leached with alcohol and followed by H <sub>2</sub> O rinses.

\*Discontinued

CONFIDENTIAL

CONFIDENTIAL

CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE VI---CONCLUDED

Sample	Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>															
	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	76
R-4460, Beryico	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	• Refluxed at 173° then leached for 2 days with alcohol.							
R-4460, Beryico	3.1	3.6	4.2	4.2	4.7	5.6	5.6	5.6	5.6	5.6	5.6	5.6	• Refluxed at 173° then leached for 5 days with alcohol.			
R-4460, Beryico	3.1	3.6	4.2	4.2	4.7	5.6	5.6	5.6	5.6	5.6	5.6	5.6	• Refluxed at 173° then leached for 5 days with alcohol.			
R-4460, Beryico	9.7	9.7	12	12	• Refluxed at 173° 1 alc rinse, 1 H <sub>2</sub> O rinse.											
R-4460, Beryico	5.8	5.8	6.9	6.9	6.9	• Refluxed at 173° 1 alc rinse, 1 H <sub>2</sub> O rinse.										
R-4460, Beryico	4.2	4.2	5.7	5.7	5.7	5.7	5.7	5.7	• Refluxed at 173° 1 alc rinse, 3 H <sub>2</sub> O rinses.							
R-4460, Beryico	4.2	4.2	5.1	5.1	5.1	5.1	5.1	5.1	• Refluxed at 173° 1 alc rinse, 3 H <sub>2</sub> O rinses.							
R-4460, Beryico	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	• Refluxed at 173° 3 alc rinses, 1 H <sub>2</sub> O rinse.							
R-4460, Beryico	4.7	4.7	6.1	6.1	6.1	6.1	6.1	6.1	• Refluxed at 173° 3 alc rinses, 1 H <sub>2</sub> O rinse.							
R-4460, Beryico	14	23	• Aqueous 5% KOH, 1 H <sub>2</sub> O rinse.													
R-4460, Beryico	9.4	33	• Aqueous 5% KOH, 1 H <sub>2</sub> O rinse.													
R-4460, Beryico	3.1	4.2	4.4	6.0	6.0	6.0	6.0	6.0	• Refluxed at 173° 4 H <sub>2</sub> O rinses.							
R-4460, Beryico	3.1	4.2	4.4	4.7	4.7	4.7	4.7	4.7	• Refluxed at 173° 4 H <sub>2</sub> O rinses.							
R-4460, Beryico	2.1	3.6	4.0	4.0	4.0	4.0	4.0	4.0	• Refluxed at 173° then leached with H <sub>2</sub> O.							
R-4460, Beryico	3.1	4.2	4.4	4.4	4.4	4.4	4.4	4.4	• Refluxed at 173° then leached with H <sub>2</sub> O.							
R-4460, Beryico	2.1	3.6	4.7	5.1	5.1	5.1	5.1	5.1	• Refluxed at 173° 1 alc rinse and then leached with H <sub>2</sub> O.							
R-4460, Beryico	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	• Refluxed at 173° 1 alc rinse and then leached with H <sub>2</sub> O.							
R-4460, Beryico	3.1	7.0	7.0	7.0	7.0	• Refluxed at 173° 1 alc rinse and then leached with H <sub>2</sub> O.										

CONFIDENTIAL

\*Discontinued.

CONFIDENTIAL



# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE VII

COMPARATIVE GAS EVOLUTION RATES OF HYDRAZINE CONTAINING  
CAUSTIC-TREATED AND UNTREATED CHROMIUM-PASSIVATED BERYLLIUM POWDER (c)

Test Conditions: Pyrex container; 90°F, 25cc Sample; 28% Be, 72% N<sub>2</sub>H<sub>4</sub>; 23% Ullage

Sample	Average Gas Evolution Rates, Duration of		Test Days	Treatment
	Untreated	Treated		
R-3951/Berylco	12.2		24	None
R-3951/Berylco	8.4		56	None
R-3951/Berylco		2.2	44	Leached for 48 hr at 90°F in alcoholic 5% KOH, three alcohol rinses.
R-3951/Berylco		1.4	93	Leached for 48 hr at 90°F in alcoholic 5% KOH, three alcohol rinses.
R-3951/Berylco		6.0	40	Refluxed at 173°F for 7 hr in alcoholic 5% KOH, three alcohol rinses.
R-3951/Berylco		10.6	24	Leached with alcohol only for 7 hr
R-3951/Berylco		2.0	60	Leached at 173°F for 20 hr in alcoholic 5% KOH, leached for 5 days with alcohol.
R-3951/Berylco		4.5	52	Leached for 64 hr at 90°F in aqueous 5% KOH, three H <sub>2</sub> O rinses.
R-3951/Berylco		2.2	52	Refluxed at 173°F for 20 hr in alcoholic 5% KOH, leached for 5 days with alcohol and then rinsed four times with H <sub>2</sub> O.
R-3951/Berylco		4.0	48	Refluxed at 173°F for 20 hr in alcoholic 5% KOH, leached for 5 days with alcohol and then rinsed four times with H <sub>2</sub> O.
R-4460/Berylco	18		12	None
R-4460/Berylco	16		12	None
R-4460/Berylco		7.0	32	Refluxed at 173°F for 20 hr in alcoholic 5% KOH, leached for 2 days with alcohol.
R-4460/Berylco		4.9	52	Refluxed at 173°F for 20 hr in alcoholic 5% KOH, leached for 5 days with alcohol.
R-4460/Berylco		4.9	52	Refluxed at 173°F for 20 hr in alcoholic 5% KOH, leached for 5 days with alcohol.

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE VII---CONCLUDED

Sample	Average Gas Evolution Rates, Duration of cc/lb-min x 10 <sup>4</sup>		Test Days	Treatment
	Untreated	Treated		
R-4460/Berylco		10.8	16	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed once with alcohol and then once with water.
R-4460/Berylco		6.5	20	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed once with alcohol and then once with water.
R-4460/Berylco		5.4	36	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed once with alcohol and then three times with water.
R-4460/Berylco		4.9	36	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed once with alcohol and then three times with water.
R-4460/Berylco		6.9	20	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed three times with alcohol and then once with water.
R-4460/Berylco		5.7	36	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed three times with alcohol and then once with water.
R-4460/Berylco		18	8	Leached for 16 hr in aqueous 5% KOH, rinsed four times with water.
R-4460/Berylco		21	8	Leached for 16 hr in aqueous 5% KOH, rinsed four times with water.
R-4460/Berylco		5.1	28	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed four times with water.
R-4460/Berylco		4.4	28	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed four times with water.
R-4460/Berylco		3.8	40	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, leached for 3 days with water.
R-4460/Berylco		4.2	40	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, leached for 3 days with water.
R-4460/Berylco		4.4	28	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed with alcohol and then leached with water for 3 days.
R-4460/Berylco		6.0	28	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed with alcohol and then leached with water for 3 days.
R-4460/Berylco		6.2	20	Refluxed at 173°F for 16 hr in alcoholic 5% KOH, rinsed with alcohol and then leached with water for 3 days.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

(C) During the course of this work, it became apparent that the best compatibilities with hydrazine were obtained with those caustic-treated powders that were thoroughly washed with water. Consequently, work was initiated to determine if the water wash alone would improve the compatibility with  $N_2H_4$ . Because the results of this water treatment were excellent, and because the treatment was much simpler, work on the caustic treatment was discontinued.

#### 2 The Effect of Water Treatment

(C) Of the five materials used to pretreat the beryllium powder, water was the most effective and practical, making it possible to meet the goal for gas evolution rate. Two procedures were used for the treatment of the powder. In the first, the powder was placed in the thimble of a Soxhlet apparatus and leached with distilled water for 4 days. The powder was then dried in a vacuum at 200°F and stored in a nitrogen atmosphere until tested for compatibility with ungelled hydrazine. Powders treated in this manner will be referred to as "leached" samples.

(C) In the second procedure the beryllium was placed directly in the distilled water, which was refluxed for 16 hr. This operation was repeated three more times with fresh water before the powder was dried and stored as in the first procedure. Powders treated in this manner will be referred to as "refluxed" samples.

(C) When four different samples of chromium-coated Berylco powder, Grade PS-98, were subjected to the leaching procedure, the gas evolution rate in ungelled hydrazine was reduced from an average of  $15 \times 10^{-4}$  cc/lb-min to an average of  $1.5 \times 10^{-4}$  cc/lb-min (range  $0.8-2.5 \times 10^{-4}$  cc/lb-min) at 90°F. These values are based on the results of eight experiments on untreated samples from four lots of powder and seven experiments with leached powder from these lots. Two refluxed samples from one of the lots had an average gas evolution rate of  $3.5 \times 10^{-4}$  cc/lb-min, over twice the average value for the leached samples (see Table VIII). When the leaching procedure was applied to a sample Brush FP-1755 powder, the average gas evolution rate for duplicate compatibility tests was  $11 \times 10^{-4}$  cc/lb-min, indicating that this type of powder is not susceptible to the water treatment used.

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

TABLE VIII

EFFECT OF WATER-TREATMENT ON COMPATIBILITY OF  
CHROMIUM-COATED BERYLLIUM POWDER WITH UNGELLED HYDRAZINE (C)  
(25-cc Samples Containing 28% Be, 23% ullage, 90°F)

<u>Powder Designation</u>	<u>Water Treatment</u>	<u>Test Duration, days</u>	<u>Gas Evolution Rate cc/lb-min x 10<sup>4</sup></u>
R-4460	None	12	18
R-4460	None	12	16
R-4460	leached	40	1.9
R-4460	leached	40	2.5
R-4460	refluxed	40	3.4
R-4460	refluxed	40	3.6
R-4630	None	12	7.3
R-4630	None	12	8.0
R-4630	leached	20	2.4
R-4635	None	4	19
R-4635	None	8	23
R-4635	leached	20	0.8
R-4635	leached	20	1.2
R-4639	None	19	14
R-4639	None	19	14
R-4639	leached	20	1.1
R-4639	leached	20	0.8

(CONFIDENTIAL TABLE)

(C) The reflux method of water treatment was examined further in three experiments with chromium-coated Berylco powder and a sample of GAC Grade 4 powder, except that changes were made in the conditions used. The reflux time was increased from 4 days to 9 days, the storage temperature for the compatibility tests was reduced from 90° to 77°F, and the beryllium content of the hydrazine was reduced from 28 to 21%. The latter two modifications were made to bring the conditions of the compatibility tests into line with those to which the goal for gas evolution rate apply. Under these conditions of treatment and compatibility testing the gas evolution rates averaged  $0.9 \times 10^{-4}$  cc/lb-min for the Berylco powder and  $1.0 \times 10^{-4}$  cc/lb-min for the GAC powder (see Table IX).

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

TABLE IX

EFFECT OF WATER-TREATMENT ON COMPATIBILITY  
OF BERYLLIUM POWDER WITH UNGELLED HYDRAZINE (C)  
(25-cc Samples Containing 21% Be, 23% ullage, 77°F)

<u>Powder Designation</u>	<u>Water Treatment</u>	<u>Test Duration, days</u>	<u>Gas Evolution Rate cc/lb-min x 10<sup>4</sup></u>
Berylco R-4635*	Refluxed 9 days	67	1.3
Berylco R-4635*	Refluxed 9 days	67	0.9
Berylco R-4635*	Refluxed 9 days	88	0.5
Berylco R-4635*	Refluxed 9 days	88	0.9
GAC Grade 4	Refluxed 9 days	67	2.0
GAC Grade 4	Refluxed 9 days	85	0.0

(CONFIDENTIAL TABLE)

\*Chromium-coated Grade PS-98

(C) Although the purpose of the chemical treatments was to leach reactive impurities from the powders or passivate active sites for catalytic hydrazine decomposition, there was a possibility that the reduced gas evolution rates effected by the treatments with alcoholic KOH and water were due instead to the presence of heavier oxide coatings on the powders. To examine this possibility, the metal contents of selected beryllium powders were determined before and after these treatments, using a eudiometric technique. These analyses showed that the treatment of chromium-coated Berylco powders with alcoholic KOH solutions caused a reduction in the average metal content from 93.7 to 91.4%. When GAC Grade 4 powder was treated with water by the reflux procedure (9 hr), its metal content was reduced from 97.5 to 93.7%. On the other hand, no loss of metal could be detected in samples of chromium-coated Berylco powder that had been water treated by the reflux method for 4-day and 9-day periods (Table X).

TABLE X

BERYLLIUM METAL CONTENT OF SELECTED WATER-TREATED  
CHROMIUM-PASSIVATED BERYLLIUM SAMPLES (U)

<u>Sample</u>	<u>Beryllium Metal Content, wt%</u>	
	<u>Before Water Treatment</u>	<u>After Water Treatment</u>
R-4630	94.4	94.3
R-4635	94.4	94.5
R-4639	93.4	93.8

(UNCLASSIFIED TABLE)

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

#### (C) Compatibility of Coated Beryllium Powders with Hydrazine

(C) The use of surface coatings were investigated as a method of increasing the compatibility of beryllium with hydrazine. Two types of coatings were an organic polysemicarbazide film, and a metallic aluminum coating.

##### 1 Polysemicarbazide Coatings

(C) In a previous program it was found that a polysemicarbazide film was the least permeable to hydrazine of a number of films tested and was the most effective in reducing the gas evolution rate of aluminum hydride-hydrazine mixtures.

(C) Two samples of beryllium powder were coated with the polysemicarbazide. Ninety-five gm of beryllium powder (R-3359) were placed in a round-bottomed flask, which was then flushed with nitrogen gas by alternately filling and evacuating the flask. The powder was then mixed with 1.75 gm (0.019 mole) of ethylene dihydrazine by stirring for 1 hr in a nitrogen atmosphere. Hexamethylene diisocyanate (3.58 gm, 0.021 moles) was added by the drop with vigorous stirring over a 1/2-hr period. A 20-gm sample was then removed from the flask and more of each reagent added as described above to increase the coating on the remaining powder. When these samples were dried and tested for compatibility with hydrazine, the gas evolution rates were observed to be excessively high (61 and 67 cc/lb-min), therefore the work with the polysemicarbazide coatings was discontinued.

##### 2 Aluminum Coatings

(C) The use of aluminum coatings to improve the chemical compatibility of beryllium powder with hydrazine was investigated. In order to evaluate coatings of this type, a fluidized bed was used. The process consisted of passing nitrogen gas saturated with triethylaluminum through a fluidized bed of heated beryllium powder where the aluminum ethyl was pyrolyzed and metallic aluminum was deposited on the beryllium powder. Although the process was not sufficiently developed to permit reliable deposition of continuous aluminum coatings on the powder, sufficient aluminum was deposited to provide an indication of the effectiveness of this treatment on the compatibility of the coated powder with hydrazine. Two samples of aluminum-coated beryllium powder (FP-1755) in ungelled hydrazine were prepared. The gas evolution rate of one of these samples was too high to measure and the second was 66 cc/lb-min. Because these rates were so high, work on aluminum-coated powders was terminated.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

#### (d) The Effect of Heat-Treatment of Beryllium Powder

(C) Because the mass spectrographic studies, described in the section on volatile impurities show that there are volatile impurities in the beryllium powder, the feasibility of heat-treating the powders prior to preparing propellants was investigated. In the first test a sample of FP-1755 powder was heated to 600°C in an argon-purged muffle furnace. The gas evolution rate of this powder in hydrazine was reduced approximately seven-fold to  $1.2 \times 10^{-4}$  cc/lb-min. However, analysis of the treated powder showed that the metallic beryllium content was reduced from 95.4% to 82.8%. This was unacceptable and was attributed to air leakage into the muffle furnace. Additional tests were then performed in which the FP-1755 powder was heated to 600°C for 4 hr under vacuum. For these powders hydrazine compatibility tests showed that in one case the gas evolution rate was decreased to about one-half that of the untreated powder, and in the second only slight improvement was shown. The loss in metallic beryllium in these samples as a result of the heat treatment was 1.3 and 3.9%, respectively. Heat treatment of alcoholic KOH treated R-4460 produced material that was more reactive than the powder was before heat treatment. The results of this investigation are summarized in Table XI.

(C) Although this investigation indicates that there may be some merit in heat-treating the beryllium powders, especially those with oxide-coatings, it was discontinued in order to pursue the more promising water-treatment described previously.

#### (e) Evaluation of Sources of Incompatibility

##### 1 Composition of Gaseous Reaction Products

(C) Analyses of the reaction products of mixtures of various beryllium powders and hydrazine were made to determine the interaction of the various beryllium powders with hydrazine. Mixtures of beryllium powder in hydrazine were placed in 60-cc ampoules, cooled to -78°C, evacuated quickly and sealed with an oxygen torch. The ampoules were stored in a constant temperature bath at 41°C for 7 days. Each ampoule was then attached to a mass spectrometer, cooled to 0°C and broken open to allow the ullage gases to enter a chamber of the mass spectrometer where the pressure and temperature were measured in a known volume. The gas samples were then analyzed. Control tests were also conducted on a beryllium sample sealed in an ampoule without hydrazine and on hydrazine sealed in an ampoule without beryllium.

CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XI  
COMPATIBILITY OF HYDRAZINE WITH HEAT-TREATED BERYLLIUM POWDERS (C)

Test Conditions: Pyrex container; 90°F; 25cc Sample, 28% Be, 72% H<sub>2</sub>; 23% Ullage

Sample	Treatment	Gas Evolution Rate, cc/lb-min x 10 <sup>-4</sup>															Content Be Metal after Treatment, %
		4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	
FP-1755** BEC	Heated to 600°C under argon.	2.6	2.1	1.5	1.5	1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.5	0.5	*	82.8	
FP-1755** BEC	Heated to 600°C under vacuum.	4.2	2.0	2.0	2.0	1.7	1.7	1.7	1.7	*						94.1	
FP-1755** BEC	Heated to 600°C under vacuum no exposure to air after testing.	7.3	5.2	4.2	3.1	2.1	*									91.6	
FP-1755** BEC	Heated to 600°C under vacuum exposed to air after testing.	37	*														
R-1460 Beryllco	Heated to 600°C under vacuum.	24	14	*													
R-1460 Beryllco	Refluxed with alcoholic KOH, extracted with alcohol and heated to 600°C under vacuum.	Too rapid to warrant calculation.															
GAC-A-88 General Astro- Metals	Heated to 600°C under vacuum.	13	5.8	3.1	*												
GAC-A-88 General Astro- Metals	Heated to 600°C under vacuum.	16	7.3	*													

\*Discontinued.  
\*\*Beryllium metal content before treatment, 95.4%.

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## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

(C) The beryllium powders which were tested are: (1) Brush FP-1755, which is used in the prototype Beryllizine; (2) Berylco R-4460, untreated; (3) R-4460, treated with alcoholic KOH for 18 hrs then extracted with ethyl alcohol; (4) R-4460, extracted with water for 3 days; (5) General Astrometals GAC 4-88, the purest beryllium powder available; and (6) R-2039 and (7) R-3359 which are similar in content of each of the analyzed impurities except aluminum which is 0.80 and 0.095, respectively. Commercial anhydrous hydrazine from one lot was used in all phases of this program.

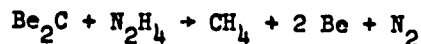
(C) The data in Table XII shows that Berylco R-4460, a chromium-passivated beryllium powder, which was extracted with water for 3 days, is more compatible with hydrazine than any of the other beryllium powders tested. The total volume of gas evolved by the water treatment is reduced to less than one-thirteenth that of the untreated R-4460 powder. Additional testing of R-4460 beryllium powder treated with alcoholic KOH indicates that this treatment also improves the chemical stability of chromium-passivated beryllium with hydrazine; however, this treatment is not as effective as the water extraction. The improvement in chemical compatibility appears to be related to the reduction in  $H_2$ ,  $CH_4$ , and  $N_2$  evolved by the alcoholic KOH-treated and water-extracted samples. The results of these tests are in agreement with the chemical compatibility tests reported in Beryllium Powder-Hydrazine Compatibility Investigations (Section III,A,3,a,(1)).

(C) Table XIII shows that Berylco R-4460 has more adsorbed gases than any of the others tested. It appears that the extraction and subsequent drying effectively remove these gases and that they have little effect on the compatibility of the treated beryllium powder in hydrazine.

(U) Analysis by vapor phase chromatography of the hydrazine in contact with the Brush FP-1755 indicated that a measurable amount of ammonia was present. Hydrazine in contact with Berylco R-2039 showed less than 0.15% ammonia. The accuracy of the vapor phase chromatograph is not sufficient to obtain a meaningful analysis of ammonia because hydrazine decomposes to produce small amounts of ammonia while it is in the chromatographic column.

#### 2 The Role of Beryllium Carbide

(C) Since methane and nitrogen were found in the gaseous reaction products of beryllium and hydrazine (see Table XII) it is believed that a carbide, probably beryllium carbide, is present. The reaction would be as follows:



COMPOSITION OF GASEOUS REACTION PRODUCTS OF BERYLLIUM AND HYDRAZINE (C)

Total Moles/g Be		Be Analysis, Percent by Weight									
Gas Analysis		Beryllio R-4460 As Received	Beryllio R-4460 Refluxed CH <sub>3</sub> OH/KOH	Beryllio R-4460 Refluxed CH <sub>3</sub> OH/KOH	Beryllio R-4460 Extracted with H <sub>2</sub> O	Beryllio R-2039 Mole % Mole/g	Beryllio R-3359 Mole % Mole/g	General Amo-Metals CAC-4-Lot 88	Control Brushes PP-1755 Only	Control Hydratins Only	
H <sub>2</sub>	222.0 x 10 <sup>-7</sup>	203.8 x 10 <sup>-7</sup>	187.5 x 10 <sup>-7</sup>	54.9 x 10 <sup>-7</sup>	15.73 x 10 <sup>-7</sup>	191.8 x 10 <sup>-7</sup>	114.90 x 10 <sup>-7</sup>	190.0 x 10 <sup>-7</sup>	15.5 x 10 <sup>-7</sup>	8.915 x 10 <sup>-7</sup>	
CH <sub>4</sub>	4.7 10.43*	23.2 47.28*	11.2 16.58*	14.0 7.55*	25.5 4.01	90.5 17300	83.8 9600	34.4 51.60*	22.8 3.53*	4.5	
H <sub>2</sub>	43.8 97.24	37.5 76.43	-	0.6 0.38	16.5 2.60	2.7 517	3.9 446	17.4 26.10	56.2 8.71	-	
H <sub>2</sub> O	50.6 112.30	30.9 79.28	88.4 130.40	85.0 45.04	55.4 8.72	6.5 124.5	11.9 1360	48.1 72.15	3.3 0.51	77.8	
O <sub>2</sub>	0.5 1.11	0.1 0.20	0.4 0.59	0.2 0.11	0.8 0.13	0.2 38	0.2 23	0.1 0.15	8.0 1.24	5.3	
CO <sub>2</sub>	0.4 0.88	T	T	-	0.2 0.04	-	0.1 11	T	0.8 0.11	0.4	
CO <sub>2</sub>	T	-	-	-	0.1 0.02	-	-	-	8.9 1.38	0.2	
HF <sub>3</sub>	-	-	-	-	0.9 0.14	T	-	-	-	10.2	
<hr/>											
BeO	1.35	0.5				0.65	1.81	1.73			
Fe	0.14	0.102				0.076	0.048	0.033			
Al	0.07	0.192				0.079	0.068	0.049			
Mg	< 0.01	0.016				0.8	0.095	0.001			
Mn		0.010				0.007	0.004	0.027			
Cr		0.013				0.010	0.008	0.001			
Si		0.065				0.005	0.005	0.03			
C	0.11	0.24				0.043	0.05	0.016			
r coated	Mo	Yes				0.62	0.47	0.015			
verage site	17	11				Mo	Mo	Mo			
(μ)						9.5	9.4	20			

\*All values of moles per gram are  $\times 10^{-7}$   
 \*\*Computed as if 20% of beryllium powder were present. Analyses of the gases not available.  
 \*\*\*This sample was heated for 10 days at 410°C.

**TABLE XIII**  
**COMPOSITION OF VOLATILE IMPURITIES OF BERYLLIUM (C)**

	Bruah FF-1755	Beryllco R-4460	Beryllco R-4460 Refluxed CH <sub>3</sub> OH/KOH	Beryllco R-4460 Water Extracted	General Astronautals GAC - Lot 88	Beryllco R-3359
TOTAL Mole/g Be	15.5 x 10 <sup>-7</sup>	84.7 x 10 <sup>-7</sup>	13.6 x 10 <sup>-7</sup>	18.9 x 10 <sup>-7</sup>	14.9 x 10 <sup>-7</sup>	32.7 x 10 <sup>-7</sup>
O <sub>2</sub> Analysis*	Mole % Mole/g	Mole % Mole/g	Mole % Mole/g	Mole % Mole/g	Mole % Mole/g	Mole % Mole/g
H <sub>2</sub>	22.8	3.53**	75.0	37.6	4.4	54.5
CH <sub>4</sub>	56.2	8.71	0.3	0.3	5.1	16.9
H <sub>2</sub>	3.3	0.51	3.2	1.8	1.8	3.5
H <sub>2</sub> O	8.0	1.24	20.1	53.6	81.6	23.4
O <sub>2</sub>	0.8	0.11	0.9	0.5	0.4	0.7
CO <sub>2</sub>	8.9	1.38	0.1	5.4	6.7	0.7

\*Volatile gases evolved by beryllium samples heated to 41°C for 10 days.  
\*\*All values of moles/gram are times 10<sup>-7</sup>.

**CONFIDENTIAL**

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

(C) In order to determine how much beryllium carbide was present in Brush FP-1755 beryllium, a 0.57-gm sample was placed in a 300-cc flask which was connected to a gas sample bulb. The system was purged with helium and 200-cc of 5N HCl was added with stirring and the resulting solution became clear and colorless, with no residue. The volume of gas evolved was measured and a sample analyzed by vapor phase chromatography.

(C) Analysis of the gas indicated that 1 gm of beryllium produced  $105.6 \times 10^{-7}$  moles of methane, which is essentially the same amount of methane produced as when the same beryllium is reacted with hydrazine ( $97.25 \times 10^{-7}$  and  $110.00 \times 10^{-7}$  moles/gram).

(C) According to the manufacturer's analysis there is 0.11% carbon present in the beryllium, which amounts to  $917.5 \times 10^{-7}$  moles/gram of beryllium. It appears that under the conditions of these tests only approximately 10% of the carbon in FP-1755 was converted to methane.

### 3 Volatile Impurities

(C) Work reported above indicated that volatile impurities are present in the beryllium powder. Two experiments were conducted to free these volatiles from beryllium and identify them. In one experiment (described in a preceding section) 8.1 gm of beryllium were placed in a glass ampoule, quickly evacuated and sealed with an oxygen torch. It was then stored at 41°C for 10 days and the evolved gas measured and analyzed. Degassing was minimized by sealing the ampoule very quickly at ambient temperature.

(C) In the other experiment 2 gm of beryllium, Brush FP-1755, were put into a 10-cc stainless-steel bomb which had been degassed under a vacuum of  $10^{-7}$  Torr while at red heat (approximately 800°C). This bomb was attached to the mass spectrometer by means of stainless-steel tubing in such a way that no organic materials (valve packings, gaskets, etc.) were involved. The bomb was then evacuated until a vacuum of  $10^{-7}$  Torr was obtained (about 20 min) then heated until it was red hot. The evolved gas was collected in an outer chamber where its quantity was measured and then passed into the mass spectrometer for analysis. The bomb was cooled to ambient temperature, evacuated to  $10^{-7}$  Torr, and the procedure repeated a second and a third time. During the third heating, gas was evolved as readily as during the first heating, indicating that only part of the volatile materials had been freed. The results of these two experiments are shown in Tables XIII and XIV.

CONFIDENTIAL

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XIV  
COMPOSITION OF VOLATILE IMPURITIES ON BERYLLIUM (BRUSH FP-1755)

Total Moles/g Be		Beryllium heated 41°C for 10 days, 15.50 x 10 <sup>-7</sup>		Beryllium heated 800°C 1st heating, 621 x 10 <sup>-7</sup>		Beryllium heated 800°C 2nd heating, 1055 x 10 <sup>-7</sup>		Beryllium heated 800°C 3rd heating, 621 x 10 <sup>-7</sup>		Total analysis of Beryllium heated 800°C, 2297 x 10 <sup>-7</sup>	
Gas Analysis	Mole %	Mole/g	Mole %	Mole/g	Mole %	Mole/g	Mole %	Mole/g	Mole %	Mole/g	Mole %
H <sub>2</sub>	22.8	3.53*	92.08	572*	98.68	1041*	99.29	617*	97.06	2229*	
CH <sub>4</sub>	56.2	8.71	-----	-----	-----	-----	-----	-----	-----	-----	
N <sub>2</sub>	3.3	0.51	1.46	9.06	0.59	6.22	0.33	2.05	0.75	17.23	
H <sub>2</sub> O	8.0	1.24	2.40	14.9	0.53	5.59	0.38	2.36	1.00	22.97	
O <sub>2</sub>	0.8	0.12	-----	-----	-----	-----	-----	-----	-----	-----	
CO <sub>2</sub>	8.0	1.38	3.89	24.16	0.20	2.11	-----	-----	1.14	26.19	
HCL	-----	-----	0.17	1.06	-----	-----	-----	-----	0.04	01	

\*All values of moles per gram are x 10<sup>-7</sup>

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

#### 4 Conclusions

(C) It is apparent from Table XII that Brush FP-1755 beryllium powder contains a carbide, probably beryllium carbide, that reacts slowly with hydrazine to produce nitrogen and methane in equal quantities. This reaction produces 94% of the pressure development observed during storage testing of FP-1755. Other beryllium powders tested contain varying amounts of beryllium carbide. The presence of beryllium carbide accounts for 75% of the pressure development in untreated Berylco R-4460-hydrazine mixtures, 34.8% when General Astro Metals GAC-4-Lot 88 is used, 10.2% when Berylco R-3359 is used, and 7.2% when Berylco R-2039 is used. It is apparent, then, that there is another reaction between some of the beryllium powders and hydrazine that produces major amounts of hydrogen and nitrogen. This may be catalytic decomposition of hydrazine but an analysis for ammonia in the hydrazine is necessary to confirm this. There is no correlation between manufacturer's analyses of impurities and the evolution of hydrogen.

(C) The Berylco R-4460 beryllium powder, after treatment with water produced less gas while in contact with hydrazine than any other beryllium tested. There was only a small quantity of methane, and this represented 37.5% of the gas evolved from the untreated powder. It is assumed that the water treatment removed nearly all of the carbide that was available to react with hydrazine.

(C) Adsorbed gases in the beryllium may produce some of the pressure development, as indicated by the gas evolved from the beryllium stored in a sealed ampoule at 41°C (see Table XIII). The gas, mostly hydrogen, released from the beryllium at high temperature probably results primarily from the reaction of water with beryllium to form  $\text{BeO} + \text{H}_2$ . The water could be formed by dehydration of metal hydroxides or basic metal oxides or could be present in a strongly adsorbed form. There is no evidence that the gases evolved from beryllium at high temperatures make a significant contribution to the pressure development which occurs when the beryllium is put in hydrazine.

#### (2) Effect of Gelling Agents on the Gas Evolution Rates of Beryllium and Hydrazine Mixtures

(C) An investigation of the gas evolution rates of mixtures of beryllium in ungelled hydrazine and mixtures of beryllium in gelled hydrazine has shown that the gelling agent affects the gas evolution rate. The results of this study are presented below.

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

(C) One of the beryllium samples selected for the studies of gas evolution was Powder GAC 4-88, manufactured by General Astro Metals Corporation. It was selected because it represented the purest commercially available beryllium powder. The gas evolution rate of a mixture of this powder and ungelled hydrazine was measured, and the results are presented in Table XV.

TABLE XV

### GAS EVOLUTION RATE OF BERYLLIUM POWDER GAC 4-88 IN UNGELLED HYDRAZINE (C)

Test Temperature 90°F  
Be 28% by weight  
Ullage 23%

Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>														
Time, days	4	8	12	16	20	24	28	32	36	40	44	48	52	56
Test No.														
6	6.4	6.4	6.4	7.5	6.4	6.4	5.5	5.5	5.5	5.4	3.9	3.9	3.9	3.9*
25	5.4	7.5	6.4	6.4	8.6	6.4	6.4	7.5	*					

(CONFIDENTIAL TABLE)

\*Discontinued

(C) The results of these tests showed that GAC 4-88 had the best chemical compatibility of any of the untreated beryllium powders tested. Because the gas evolution rates of this powder were lower than the gas evolution rates of the prototype Beryllizine using Brush Beryllium 1755, a laboratory batch of Beryllizine-21 was prepared with GAC 4-88 to determine if the gas evolution rates would be reduced. No improvement was observed. The results of the gas evolution rate measurements made on this material are presented in Table XVI.

TABLE XVI

### GAS EVOLUTION RATES OF BERYLLIZINE-21 (C)

Test Temperature 77°F  
Be (GAC 4-88) 21% by weight  
Ullage 10%

Gas Evolution Rate,  
cc/lb-min x 10<sup>4</sup>

1 week

15

1 month

10

(CONFIDENTIAL TABLE)

# CONFIDENTIAL

• Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

(C) Comparison of the gas evolution rates of the mixture of beryllium in ungelled hydrazine and of the mixture of beryllium in gelled hydrazine showed that the gas evolution rate of the gelled mixture was higher. Two explanations of the observed results were considered. First, the surface of the beryllium powder was abraded during the mixing procedure, thus exposing active sites on the beryllium powder. These active sites were then catalyzing the decomposition of the hydrazine. A second possible explanation was that the Carbopol-940 was entering into the reaction between the beryllium and hydrazine and increasing the gas evolution rate.

(C) A series of tests was conducted to determine which of the two explanations was correct. Two sets of beryllium-containing gels were prepared. A Carbopol-940 concentration of 0.25% was selected to provide thin gels so that the beryllium powder could be easily dispersed. In the first case, a gel was prepared and the beryllium powder GAC 4-88 was then added to the gel with a minimum of stirring. This technique was used so that the beryllium powder would not be exposed to any shearing which could abrade its surface. In the second case, the beryllium powder was added to the hydrazine, and then the Carbopol-940 was gradually added. This mixture was vigorously sheared during the preparation. The results of gas evolution rate measurements made on these samples are presented in Table XVII, along with data on mixtures of beryllium in ungelled hydrazine.

TABLE XVII

EFFECT OF SHEARING ON THE GAS EVOLUTION  
RATE OF BERYLLIUM IN GELLED HYDRAZINE (C)

		Test Temperature		90°F													
		Be (GAC 4-88)		28% by weight													
		Ullage		23%													
		Gelling Agent		Carbopol-940, 0.25% by weight													
		Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>															
Time, days	4	8	12	16	20	24	28	32	36	40	44	48	52	56			
<u>Sample</u>																	
Unsheared Gel	26	23	23	24	*												
Unsheared Gel	35	29	*Sample broke during venting.														
Sheared Gel	48	42	42	42	*Sample terminated because of bulk growth into the manometer.												
Sheared Gel	32	35	35	34	24	*											
Be + N <sub>2</sub> H <sub>4</sub> (not gelled)	6.4	6.4	6.4	7.5	6.4	6.4	5.5	5.5	5.5	5.4	3.9	3.9	3.9	3.9*			
Be + N <sub>2</sub> H <sub>4</sub> (not gelled)	5.4	7.5	6.4	6.4	8.6	6.4	6.4	7.5	*								

\*Discontinued

(CONFIDENTIAL TABLE)



## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

(C) The results show that shearing does not affect the gas evolution rates in gelled hydrazine containing beryllium powder. The high gas-evolution rates of the sheared and unsheared beryllium powders in hydrazine gelled with Carbopol-940, compared with the gas evolution rate of the same beryllium powder in ungelled hydrazine, clearly show that Carbopol-940 is an important factor in the chemical instability of this gelled mixture.

(C) In view of these results, another series of tests was conducted to determine if they could be confirmed, and to investigate the effect on the gas evolution rate of several new gelling agents prepared by Aerojet-General Corporation during a current IR&D program.

(C) Beryllizine and hydrazine gelled with Carbopol-940 were prepared as controls. Equal concentrations of gelling agents were used based on equivalent weights. The beryllium powder selected was R-4460, a chromium-coated material manufactured by the Beryllium Corporation. The powder was pretreated by water-leaching and vacuum-drying. The results of this test series are presented in Table XVIII. The results show that mixtures of beryllium with hydrazine gelled with Carbopol-940 have substantially higher gas evolution rates than do the mixtures of beryllium and ungelled hydrazine.

(C) One of the Aerojet-General-developed gelling agents, polyhydrazine acrylate, was not effective in reducing the gas evolution rate. The results also show that gelling agents CPAA-12 and CP\* were effective. The gas evolution rates of these mixtures of beryllium and gelled hydrazine are comparable to those of mixtures of the same beryllium in ungelled hydrazine.

#### b. Effect of Beryllium Powders on Gel Formation

(C) As previously mentioned, the gelling capacity of Carbopol-940 in hydrazine is adversely affected by electrolytes which may be introduced into the propellant by the metal powder. The introduction of these electrolytes can be detected by measuring the change in the dielectric constant of the hydrazine that has been in contact with the powder. Good quality hydrazine has a dielectric constant between 45 and 48. Previous work has shown that if the metal powder introduces sufficient electrolytes to increase the dielectric constant to a value greater than 55, the gelling capacity of Carbopol is seriously affected. Consequently, the dielectric constant of the hydrazine which has been in contact with various types of untreated and treated beryllium powder has been measured.

\*CP and CPAA-12 are derivatives of cross-linked polyacrylic acid prepared by the Aerojet-General Corporation.

CONFIDENTIAL

Report AFRPL-TR-65-230

TABLE XVIII  
EFFECT OF GELLING AGENTS ON THE GAS EVOLUTION RATES OF MIXTURES  
OF BERYLLIUM AND HYDRAZINE (C)

Sample	Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>									
	8	12	16	20	24	28	32	36	40	44
Test Temperature 90°F										
Be (B-1160, water extracted) 20% by weight										
Ullage										
Be + Ungelled Hydrazine	0	1.5	1.9	1.9	1.9	1.9	1.9	1.9	1.9	•
Be + Ungelled Hydrazine	0	3.1	2.5	2.5	2.5	2.5	2.5	2.5	2.5	•
Be + Ungelled Hydrazine	0	3.1	3.1	2.9	2.6	3.1	4.0	4.2	2.1	2.5
Be + Ungelled Hydrazine	0	2.9	2.9	2.9	2.3	2.6	3.1	3.6	2.3	3.3
Be + Gelled Hydrazine	24	26	26	26	24	•	•	•	•	•
Gelling Agent: 0.25% Carbopol-940	19	24	24	24	24	•	•	•	•	•
Be + Gelled Hydrazine	20	24	24	24	24	•	•	•	•	•
Gelling Agent: 0.25% Carbopol-940	18	22	22	22	24	•	•	•	•	•
Be + Gelled Hydrazine	0.5	2.5	2.5	2.5	2.9	2.3	2.6	2.6	2.3	2.5
Gelling Agent: 0.87% CPA-12	0.5	2.3	2.3	2.3	1.8	2.3	2.1	2.3	1.5	2.9
Be + Gelled Hydrazine	0.8	2.6	2.6	2.6	2.9	3.4	2.3	2.1	2.6	3.1
Gelling Agent: 0.87% CPA-12	1.3	2.1	2.1	2.1	1.2	1.8	1.2	1.5	1.5	1.8
Control (No Be)	2.5	2.1	2.1	2.1	1.3	1.8	1.2	1.5	1.5	1.6
Hydrazine Gelling Agent: 0.25% Carbopol-940										
Same										

Discontinued.

CONFIDENTIAL

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Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

(C) To investigate the effect of beryllium on the dielectric constant of hydrazine a 5-gm sample of FP-1755 beryllium was suspended in 100 ml of neat hydrazine having a dielectric constant of 45; the mixture was stored in the oscillometer cell for 17 days. Periodic measurements made during this time showed that the dielectric constant remained within 0.5 units of the original value, and it was concluded that no leachable ionic species were present on the powder. Because this observation was not in agreement with those made earlier at the Air Force Rocket Propulsion Laboratory and at Aerojet, the experiment was repeated using 21% by weight of FP-1755 beryllium in contact with hydrazine having a dielectric constant of 46. After five days of contact the dielectric constant was 48. From this result and from the previous work it was concluded that FP-1755 beryllium powder did not adversely affect the dielectric constant of hydrazine.

(C) The effect of the powders discussed above and four other untreated beryllium powders on the dielectric constant of hydrazine is tabulated in Table XIX.

(C) The results presented in Table XIX show that FP-1755 and GAC 4-88 beryllium powders do not adversely affect the dielectric constant of hydrazine. However, the results of tests with the chromium-coated beryllium powder from Berylco indicate that these powders may adversely affect the dielectric constant of hydrazine.

TABLE XIX

### DIELECTRIC CONSTANT OF HYDRAZINE AFTER CONTACT WITH BERYLLIUM POWDERS (C)

<u>Sample</u>	<u>Contact Time, days</u>	<u>Dielectric Constant</u>
FP-1755, BBC	17	45.4
FP-1755, BBC	5	48.3
4-88, GAC	5	51.2
R-3951, Berylco	56	55
R-2407, Berylco	56	77.5
R-2508, Berylco	56	77.5

(CONFIDENTIAL TABLE)

## **CONFIDENTIAL**

Report AFRPL-TR-66-230

### **III, A, Chemical Laboratory Investigations (cont.)**

(C) It has been found that hydrazine-soluble electrolytes can be removed by water washing the chromium-coated powder before adding it to the hydrazine. For example, hydrazine exposed for 48 days to beryllium powder R-3951 which had been water washed had a dielectric constant of 47.5. The use of water to remove electrolytes from caustic-treated beryllium powders has also been demonstrated.

(C) No correlation has been found between gas evolution rates and the dielectric constant of hydrazine exposed to powders. It does not appear to be difficult to obtain or prepare beryllium powders which will not adversely affect the dielectric constant of hydrazine.

**CONFIDENTIAL**

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

### c. Development of Prototype Fuels

#### (1) Preparation of Prototype Fuels

(U) In order to provide fuels suitable for engineering investigations prior to the establishment of the final Beryllizine formulations, prototype Beryllizine-21 and Beryllizine-28 formulations were developed early in the program. The objective was to prepare fuels meeting the requirements of the tentative specification (see Appendix C) for yield stress, density, viscosity, and smoothness. However, in view of the use to be made of the prototype fuels, the storage life did not have to exceed one month.

#### (a) Components

(C) The components of the prototype fuels were hydrazine meeting the requirements of specifications MIL-P-26536 for hydrazine and MIL-P-27412 (USAF) for Alumizine-43G; beryllium powder FP-1755 manufactured by the Brush Beryllium Co.; and Carbopol-940, a polyacrylic acid gelling agent manufactured by the B. F. Goodrich Chemical Company. Specification MIL-P-27412 (USAF) requires that the water content and dielectric constant of the hydrazine not exceed 1.0% and 47, respectively. Beryllium powder FP-1755 was selected for the prototype fuel because previous work at Aerojet had shown that Beryllizine prepared with this material was stable for at least a month and that its flow properties were similar to those of Alumizine. Carbopol-940 was chosen because it was the most advanced gelling agent available at the time for hydrazine-based heterogeneous fuels.

(C) From the experience and results obtained during this program involving the preparation and testing of prototype Beryllizine-21 and Beryllizine-28, the respective Carbopol concentrations of 0.48 and 0.45% have been found to produce fuel having a yield stress value of 1500 dynes/cm<sup>2</sup>. This information is based on results from 41 batches of prototype propellant that were prepared according to the approved standard mixing procedure (see Appendix D). Consequently, the recommended Carbopol-940 concentration for the prototype Beryllizine-21 is 0.48%, and the recommended concentration of Carbopol-940 in Beryllizine-28 is 0.45%. The results of mixing prototype Beryllizine-21 and -28 presented in Tables XX and XXI support the two recommended concentrations.

#### (b) Propellant Processing Procedure

(U) The properties and rheological characteristics of gelled heterogeneous fuels are dependent on the manner in which the components are incorporated into the formulation. The yield stress and

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# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XX

SUMMARY OF PROTOTYPE BERYLLIZINE-21 BATCHES (C)

<u>Batch*</u>	<u>Carbopol-940 Conc., percent</u>	<u>Yield Stress, dynes/cm<sup>2</sup> (77°F)</u>	<u>Density, gm/cc (77°F) (1.114 theo)</u>
<u>1/2 GAL MIXER</u>			
LP-334	0.80	1850	1.110
LP-335	0.60	1500	1.112
LP-336	0.55	1400	1.112
LP-342	0.60	1550	1.111
LP-343	0.60	1550	1.112
LP-344	0.60	1750	1.111
LP-345	0.60	1600	1.112
LP-346	0.60	1500	1.114
LP-347	0.60	1500	1.107
LP-348	0.60	1550	1.111
LP-349	0.60	1000	1.108
LP-350	0.60	2000	1.113
LP-351	0.53	1250	1.114
LP-352	0.45	1000	1.113
LP-353	0.35	800	1.115
LP-354	0.20	350	1.115
<u>3-GAL MIXER</u>			
3LP-2	0.60	1600	1.118
3LP-3	0.55	1600	1.115
3LP-4	0.55	1550	1.111
3LP-5	0.52	1600	1.116
3LP-6	0.48	1550	1.113
3LP-7	0.48	1450	1.112
3LP-8	0.48	1350	1.112
3LP-9	0.49	1500	1.111
3LP-10	0.49	1500	1.111
3LP-11	0.49	1600	1.111
3LP-12	0.49	1350	1.115
3LP-14**	0.60	2050	1.114
3LP-15***	0.49	1900	1.111
3LP-25	0.49	1300	1.114

\*The batches of prototype propellant are ordinarily prepared using beryllium powder FP-1755, a 17-μ powder from Brush Beryllium Company.

\*\*Made with FP-555, 5-μ powder from Brush Beryllium Company.

\*\*\*Made with chromium-coated powder, R-4630, from Beryllium Corporation.

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XXI

SUMMARY OF PROTOTYPE BERYLLIZINE-28 BATCHES (C)

<u>Batch*</u>	<u>Carbopol-940 Conc., percent</u>	<u>Yield Stress, dynes/cm<sup>2</sup> (77°F)</u>	<u>Density, gm/cc (77°F) (1.114 theo)</u>
<u>1/2-GAL MIXER</u>			
LP-355	0.50	1900	1.158
LP-356	0.45	1400	1.155
LP-358	0.49	1900	1.155
<u>3-GAL MIXER</u>			
3LP-16	0.45	1600	1.154
3LP-24	0.45	1450	1.155
3LP-26	0.45	1350	1.152
3LP-27	0.45	1600	1.154
3LP-28	0.45	1200	1.152
3LP-29	0.46	1400	1.145
3LP-30	0.46	1300	1.149
3LP-31	0.46	1050	1.149

\*All batches were made with Beryllium Powder FP-1755 from  
Brush Beryllium Company.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

rheological properties are particularly susceptible to the rate and duration of shear applied to the fuel during preparation. It is mandatory, in view of this, that during the development of a heterogeneous fuel a standard mixing procedure is developed and put into use to reduce the number of variables that influence the properties of the product.

(U) Nineteen 1/2-gal batches and twenty-two 3-gal batches of prototype Beryllizine were prepared during the course of the program for use in both propellant and engineering investigations. The 1/2-gal batches were prepared in the laboratory mixer shown in Figure 2. The mixer used for the 3-gal batches is a scaled up version of the laboratory mixer.

(C) The mixing procedure used for the first eight 1/2-gal batches was the standard procedure developed previously for Alumizine-43G. From this work it became apparent that the low yield stress of these batches (850-1250 dynes/cm<sup>2</sup>, rising sphere method) was due to excessive shear in the mixer and that a change in the impeller speed during the final dispersion step of the mixing procedure was required to attain the desired yield stress in the product. The impeller speed was reduced to correspond with a reduction in batch size from 2700 ml for Alumizine to 2000 ml for Beryllizine. The new speed was calculated in accordance with the equation  $E = nD^2$ , which relates the total shear energy, E, to viscosity,  $\eta$ , and shear rate, D. Because the viscosities of Beryllizine and Alumizine are equal, and the shear rate is proportional to mixer speed, rpm, the total shear energy input was corrected for batch size by means of the following equation:

$$\frac{\text{Volume of Alumizine}}{\text{Volume of Beryllizine}} = \frac{(\text{rpm for Alumizine})^2}{(\text{rpm for Beryllizine})^2}$$

(U) Based on this equation the mixer speed was reduced from 1400 to 1200 rpm. Batches of fuel prepared after this change was made had yield stress values in the required range and did not display growth in yield stress during subsequent storage. The standard laboratory processing procedure for Alumizine was modified to reflect the changes in batch size and impeller speed and is included in this report (Appendix D) as the standard Laboratory Processing Procedure for Beryllizine.

#### (2) Characterization of Prototype Beryllizine-21

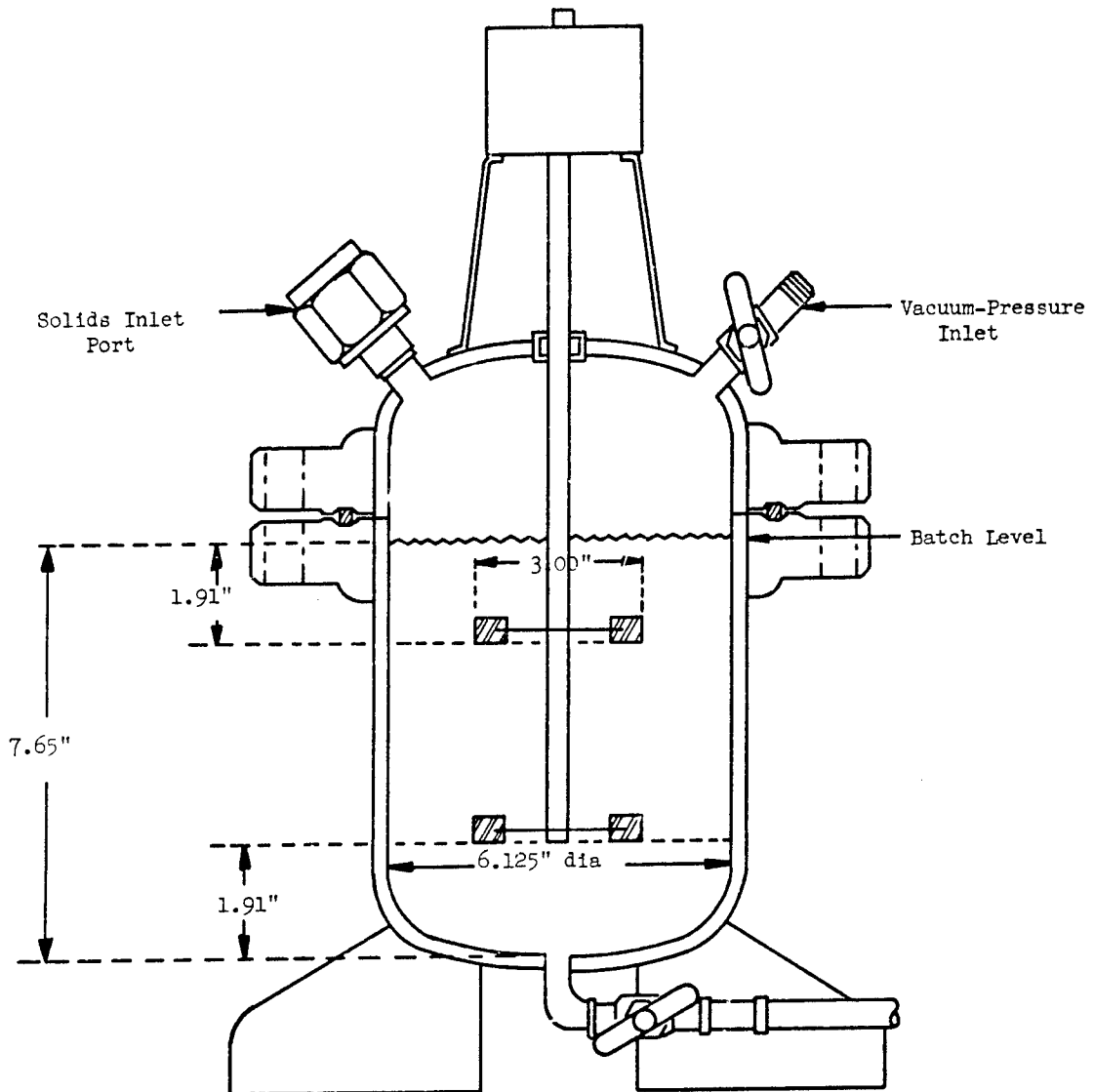
##### (a) Mechanical Stability

(C) In order to determine the relationship between yield stress and mechanical stability, samples of Beryllizine-21 having yield stress values from 350 to 1200 dynes/cm<sup>2</sup> were tested for mechanical stability under ambient laboratory conditions. These tests showed that two samples of a preparation having a yield stress of 900 dynes/cm<sup>2</sup>



**CONFIDENTIAL**

Report AFRPL-TR-66-230



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Figure 2. 1/2-Gallon Laboratory Mixer

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# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

were stable for seven weeks, three weeks longer than the minimum requirement for the prototype fuel; that two samples of this material were stable for more than three months; and that a preparation having a yield stress of 1250 dynes/cm<sup>2</sup> had densities of top and bottom samples differing by only 0.003 gm/cc after 9 months of ambient storage. This difference is within the experimental error of the density measurements. Beryllizine having a yield stress of only 350 dynes/cm<sup>2</sup> was not stable overnight.

(C) Four batches of fuel having yield stress values ranging from 350 to 1250 dynes/cm<sup>2</sup> were subjected to acceleration for 30 min at 18 g, and three of these were subjected to vibration tests. The data presented in Table XXII show that all the samples were mechanically stable to the acceleration and vibration conditions specified, except that the preparation with the lowest yield stress could not be vibration-tested because of inadequate gel structure. The Carbopol concentration of this preparation was 0.20 percent.

TABLE XXII

### STABILITY OF PROTOTYPE BERYLLIZINE-21 TO LONGITUDINAL ACCELERATION AND VIBRATION (C)

Preparation	Yield Stress, dynes/cm <sup>2</sup>	Initial Density, gm/cc	Position of Specimen	Density of Specimens after Testing, gm/cc (77°F)			
				Acceleration Testing (30 min at 18g)		Vibration Testing	
				Sample 1	Sample 2	Sample 1	Sample 2
LP-351	1250	1.114	Top	1.112	1.115	1.104	1.107
			Bottom	1.111	1.115	1.100	1.107
LP-352	1000	1.113	Top	1.110	1.112	1.110	1.105
			Bottom	1.112	1.113	1.111	1.111
LP-353	800	1.115	Top	1.112	1.112	1.122	1.114
			Bottom	1.113	1.116	1.115	*
LP-354	350	1.115	Top	1.114	1.113	Fuel unstable overnight not tested.	
			Bottom	1.118	1.111		

(CONFIDENTIAL TABLE)

\* Specimen damaged during transfer

# CONFIDENTIAL

Report AFRPL-TK-66-230

## III, A, Chemical Laboratory Investigations (cont.)

### (b) Chemical Stability

(U) The samples stored for long-term mechanical stability tests showed evidence of high rates of gas evolution and bulk growth and many were destroyed to prevent the containers from breaking. In order to obtain quantitative information on the chemical stability of the prototype fuel, gas evolution and bulk growth rate measurements were made for periods up to one month.

(C) The gas evolution rates of six samples from four batches of prototype fuel were determined in the apparatus similar to that shown in Figure 1. The sample size was 100 cc and the ullage was 10%. The data summarized in Table XXIII show that at 77°F the gas evolution rates averaged  $11 \times 10^{-4}$  cc/lb-min in those experiments that were run for 30-31 days. The extremes of the observed rates were  $9 \times 10^{-4}$  cc/lb-min in a 15-day experiment and  $22 \times 10^{-4}$  in a 17-day experiment, the overall average being  $12 \times 10^{-4}$ .

TABLE XXIII

GAS EVOLUTION RATES OF BERYLLIZINE-21 (77°F) (U)

<u>Preparation</u>	<u>Test Period, days</u>	<u>Gas Evolution Rate, cc/lb-min <math>\times 10^4</math></u>
LP-317		
Sample 1	31	10
Sample 2	21	12
LP-323	15	9
LP-326		
Sample 1	30	11
Sample 2	30	11
LP-334	17	22

(CONFIDENTIAL TABLE)

In another experiment, duplicate samples of one-month-old prototype fuel were stored in the compatibility apparatus for 30 days. During this period the rates were relatively constant, as shown in Table XXIV, and of the same order of magnitude as those summarized in Table XXIII. The data indicate that the gas evolution rates for the prototype fuel were greater than the program goal ( $1.5 \times 10^{-4}$  cc/lb-min) by a factor of 6 to 14.

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

TABLE XXIV

CHEMICAL STABILITY TESTS ON ONE-MONTH-OLD  
PROTOTYPE BERYLLIZINE-21 (77°F) (U)

Age, one month plus days	Gas Evolution Rate, cc/lb-min x 10 <sup>4</sup>		Bulk Growth Rate, %/min x 10 <sup>6</sup>	
	Sample 1	Sample 2	Sample 1	Sample 2
1	8	13	51	62
6	11	13	33	37
11	10	10	23	24
30	10	11	20	24

(CONFIDENTIAL TABLE)

(C) Bulk growth measurements were made in an apparatus identical to that used for the gas evolution rate measurements. In this case, however, the space above the sample was filled with mineral oil. The increase in fuel volume was computed from data on pressure rise and converted to a percentage of the sample volume per minute of the observation period. The results, which are summarized in Table XXV, show that the rate varied from  $75 \times 10^{-6}$  %/min at 2 days to  $6-8 \times 10^{-6}$  %/min at about one month. Data for three of the samples show that the rate decreased markedly during the period of storage. Similar results were obtained with two additional samples of one-month-old fuel that were stored in the bulk growth apparatus for 30 days (see Table XXIV). In these experiments the rate decreased from an average of  $56 \times 10^{-6}$  %/min on the first day to an average rate on the 30th day of  $22 \times 10^{-6}$  %/min. Consideration of all the rate data indicates that the highest rates, which were observed during the first week, were about 1.5 times the goal after about a month of storage. The samples stored during their second month of age displayed bulk growth rates intermediate between these values.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

TABLE XXV

BULK GROWTH RATES OF BERYLLIZINE-21 (77°F) (U)

<u>Preparation</u>	<u>Age of Sample, days</u>	<u>Bulk Growth Rate, %/min x 10<sup>6</sup></u>
LP-317	6	41
LP-318	8	45
	30	8
LP-323	7	47
	21	7
	27	6
LP-334	2	75
	10	25

(CONFIDENTIAL TABLE)

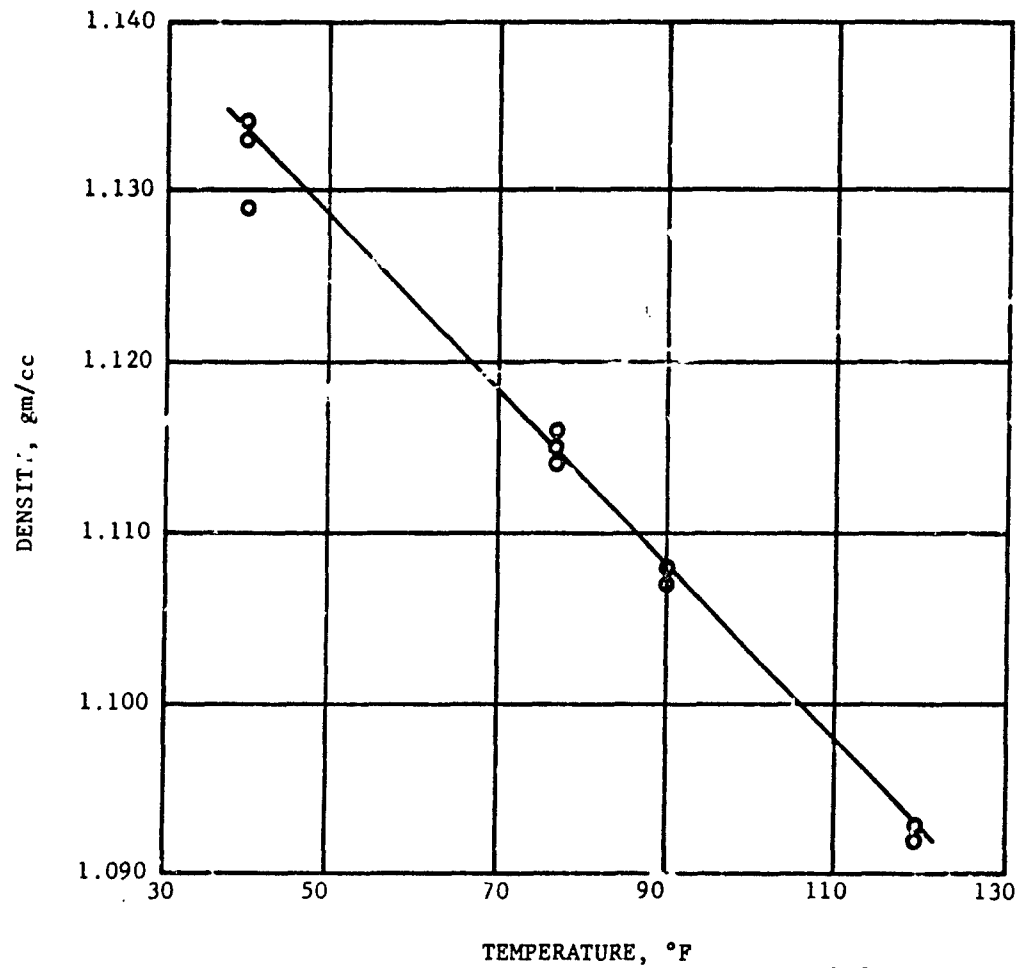
#### (c) Physical Properties

(C) Density measurements and three types of viscosity measurements were made on the prototype fuel. The former were made by the pycnometric method specified in MIL-P-27412 (USAF) for Alumizine-43G. The relation of density to temperature was determined for a sample having a yield stress of 1600 dynes/cm<sup>2</sup>. Triplicate measurements were made at 40°, 77°, 90° and 120°F. Each sample was conditioned at the test temperature for approximately 2 hr before volumetric adjustments were made according to the standard procedure for the method. The data plotted in Figure 3 show a negative slope, computed by the method of least squares, equivalent to a rate of change in the density of  $5.4 \times 10^{-4}$  gm/cc per °F ( $\beta = 4.6 \times 10^{-4}$  °F<sup>-1</sup>). The average density at 77°F was 1.115 gm/cc compared to 1.114 gm/cc, the calculated value.

(C) Characteristic flow curves for prototype Beryllizine-21 having yield stress values of 1400 and 1800 dynes/cm<sup>2</sup> were prepared from data obtained with the ASTM D-1092 viscometer. These are presented in Figure 4 along with similar curves for Alumizine-43G having yield stress values of 1400 and 1900 dynes/cm<sup>2</sup>, and show that the two fuels have comparable laminar-flow behavior in the range of shear rates from 5 to 10,000 sec<sup>-1</sup>.

**CONFIDENTIAL**

Report AFRPL-TR-66-230



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Figure 3. Density of Prototype Beryllizine-21 (U)

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Report, AFRPL-TR-66-250

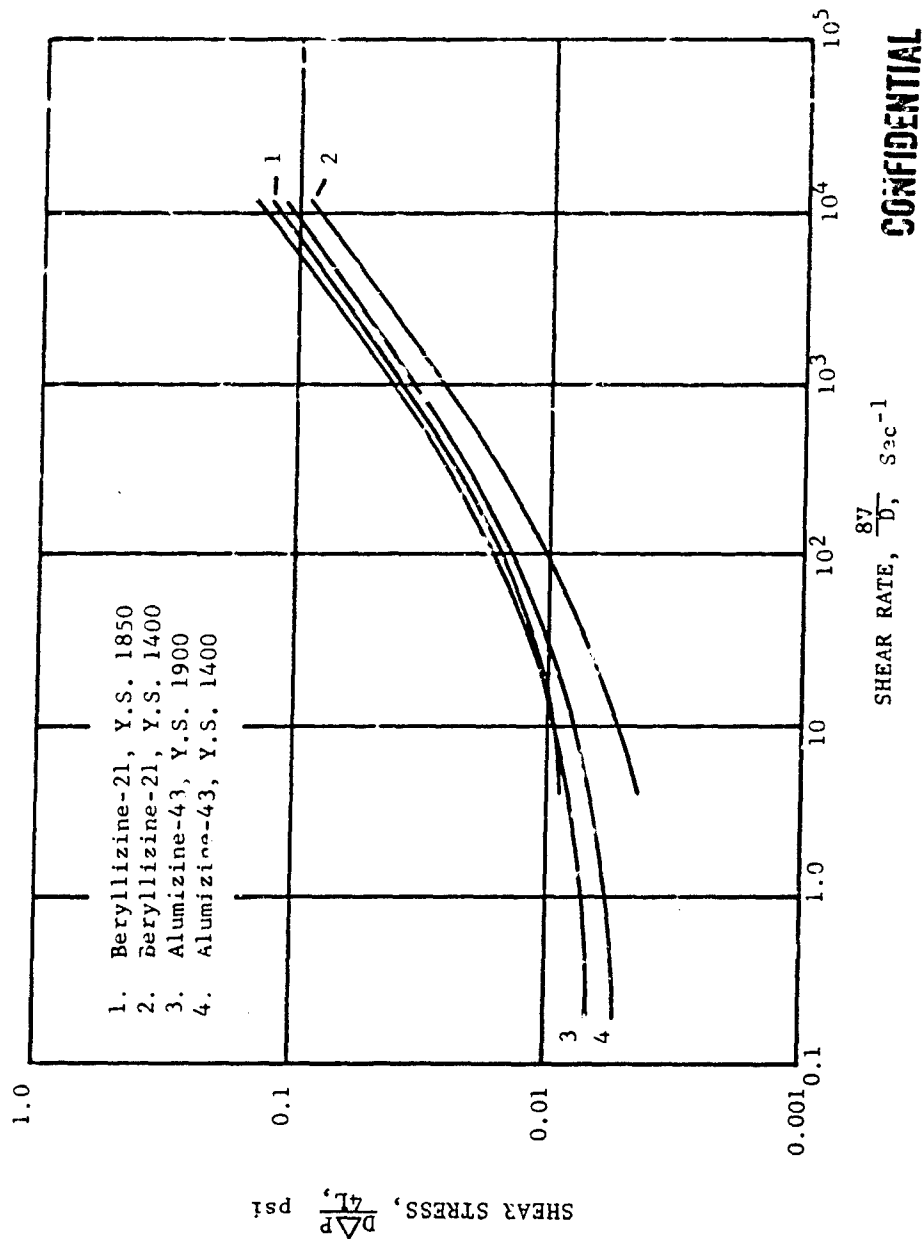


Figure 4. Characteristic Flow Curves from Measurements with ASTM D-1092 Capillary Viscometer at 17°F (U)

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Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigations (cont.)

(C) Of eleven batches of fuel examined with the Brookfield viscometer, two failed to fall within the limits of the Brookfield limiting-flow characteristic described in the military specification for Alumizine-43G. These had the lowest and highest yield stress values of the group, 800 and 1850 dynes/cm<sup>2</sup>, as shown in Figure 5. It is apparent from the figure that Beryllizine-21 having yield stress values of 1400-1500 dynes/cm<sup>2</sup> tends to approach the upper limit established previously for Alumizine-43G.

(C) Data on the viscosity of prototype Beryllizine-21 at high shear (17,300 sec<sup>-1</sup>), which were obtained with the Ferranti-Shirley viscometer, are shown in Figure 6. The line relating the high-shear viscosity to the yield stress of Beryllizine has a slope somewhat higher than that of the comparable line for Alumizine-43G, which is included in the figure for comparison. The high shear viscosity of the sample of Beryllizine-21 used for the measurements is higher than the average for Alumizine-43G. However, in the range of yield stress values below 1600 dynes/cm<sup>2</sup> the high shear viscosities of the two fuels are not significantly different because below this value the upper limit of the 95% confidence prediction interval for Alumizine-43G is higher than the line relating the high shear viscosity of the Beryllizine-21 to yield stress.

#### (3) Effect of Alternate Beryllium Powders

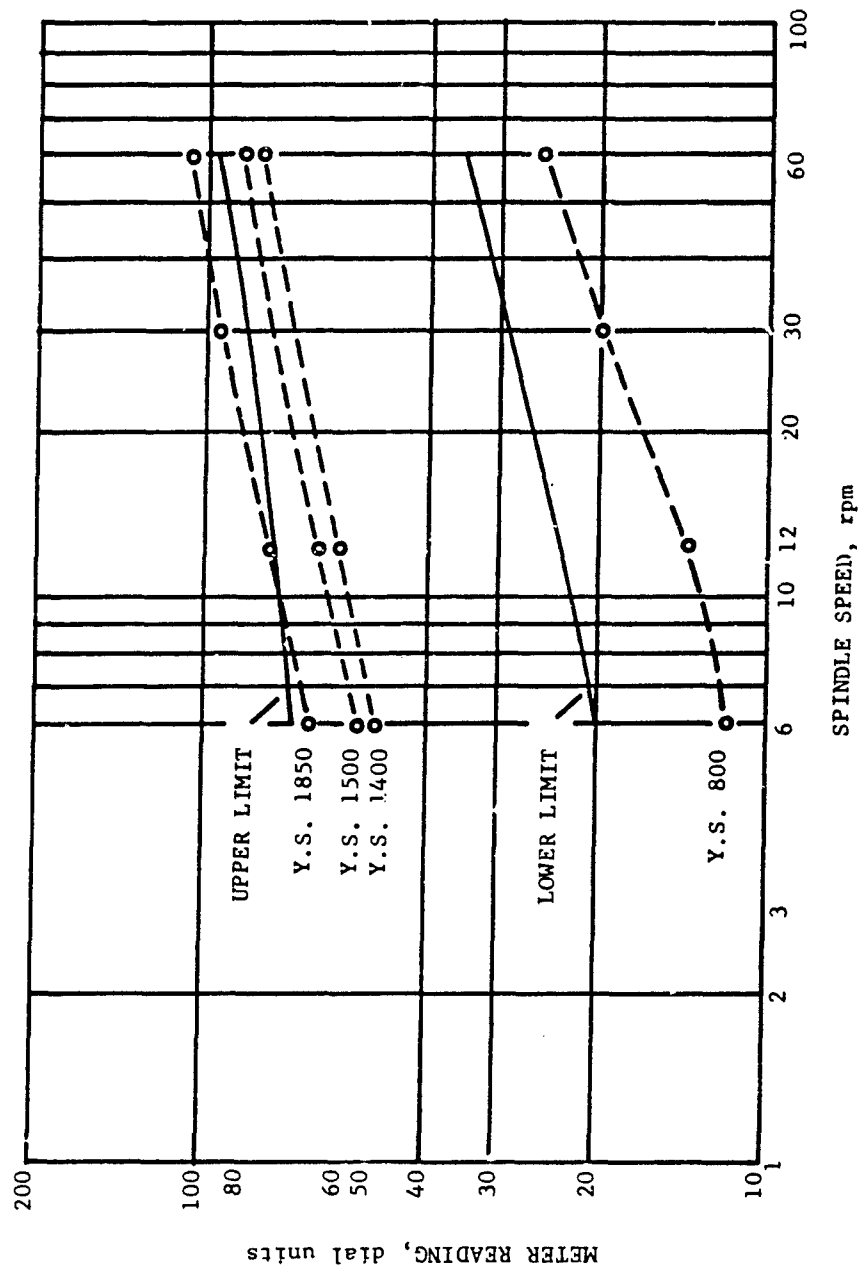
(C) Two mixes of Beryllizine-21 were made to evaluate chromium-passivated and fine-particle beryllium powders in chemical and engineering tests, and to compare them with the Brush Beryllium Company FP-555, a 5-μ powder from the Brush Beryllium Company, and R-4630, a 12-μ chromium-passivated powder from the Beryllium Corporation.

(C) Before the two powders were used to make Beryllizine, the dielectric constant of a hydrazine wash from each powder was measured in order to provide assurance that gelled fuel could be prepared with Carbopol-940. The dielectric constant of the hydrazine wash was 61 for FP-555 and 50 for the chromium-passivated R-4630. The dielectric constant of the neat hydrazine was 45 before use. Because aluminum powder and the beryllium powder FP-1755 have been found suitable for gel preparation when the dielectric constant of a hydrazine wash is in the range from 48.5 to 52, it was evident that the FP-555 (N<sub>2</sub>H<sub>4</sub> wash,  $\epsilon = 61$ ) would require an adjustment in Carbopol concentration to prepare Beryllizine-21 that would meet the yield stress specifications. The chromium-passivated R-4630 required no adjustment in the Carbopol concentration for it to be used in making prototype fuel. The results of mixing Beryllizine made with these two powders are presented in Table XX, Items 3LP-14 and 3LP-15. The high yield stress values for these fuels were unexpected and are unexplained other than they may be attributed to the high adsorption of hydrazine on the small-size powders having high surface area.



CONFIDENTIAL

Report AFRPL-TR-66-230



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Figure 5. Brookfield Limiting Flow Characteristic for Beryllizine-Cl (77°F) (U)

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Report AFRPL-TR-66-230

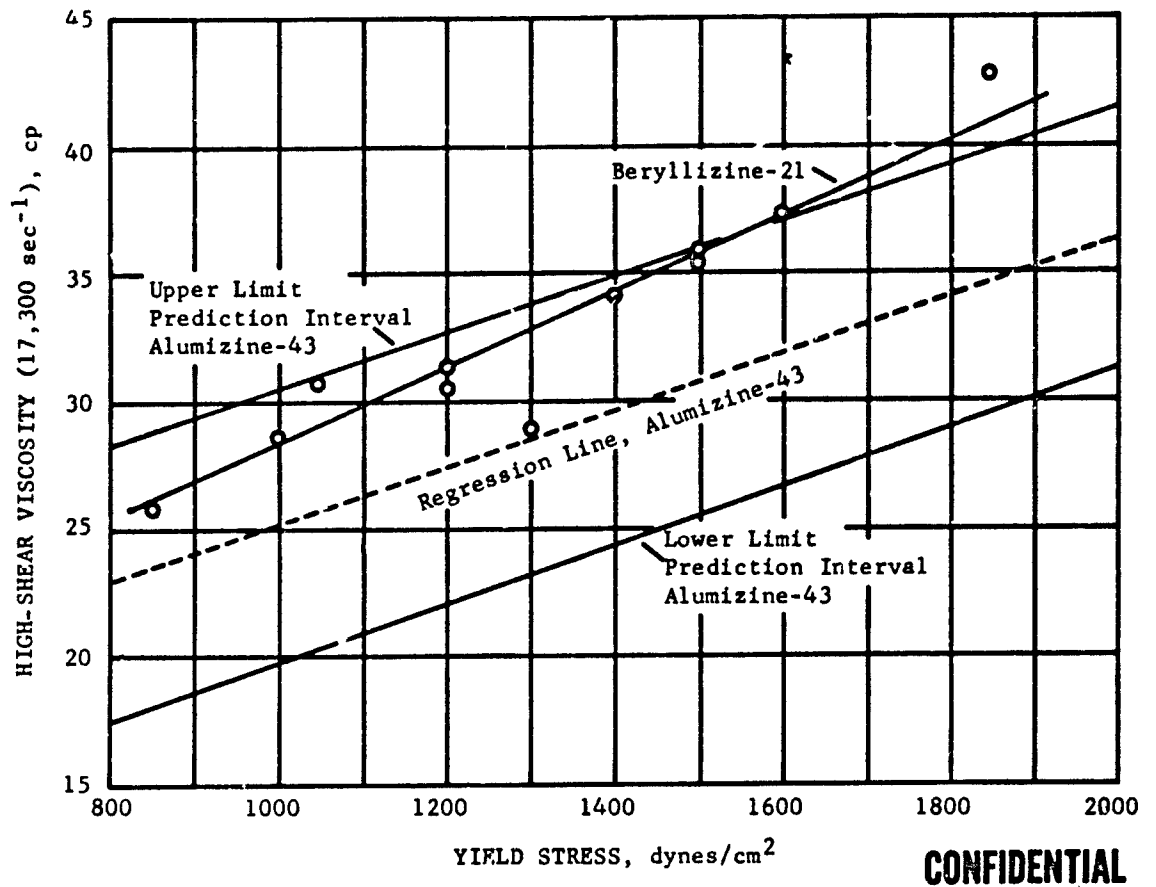


Figure 6. Relationship of High Shear Viscosity to Yield Stress (77°F) (U)

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Report AFRPL-TR-66-230

## III, A, Chemical Laboratory Investigations (cont.)

(C) The gas evolution rate of the fuel prepared with the powder FP-555 (5 $\mu$ , Brush Beryllium Company) was  $41 \times 10^{-4}$  cc/lb-min at 3 days, and this rate was too high to permit continued storage of samples. Consequently, this experimental fuel is unacceptable for even short-term storage.

(C) The gas evolution rate of the Beryllizine made with these powders was measured according to the standard procedure, and the data are presented in Table XXVI. These results indicate that the fuel prepared with chromium-passivated R-4630 has a 2-wk gas evolution rate averaging  $6.0 \times 10^{-4}$  cc/lb-min and is more stable than the prototype Beryllizine-21 (containing FP-1755), which has a typical 2- to 4-wk rate of  $10-12 \times 10^{-4}$  cc/lb-min.

TABLE XXVI

CHEMICAL STABILITY TESTS ON EXPERIMENTAL BERYLLIZINE-21  
MADE WITH THE Be POWDERS FP-555 AND Cr-COATED R-4630 (C)

Propellant Identification	Elapsed Time, days	Gas Evolution Rate, cc/lb-min $\times 10^4$ (77°F)		
		Sample 1	Sample 2	Sample 3
3LP-14*	3	41.0	41.0	34.3
(Samples grew out of apparatus, tests stopped)				
3LP-15**	3	6.1	4.2	2.5
	7	5.3	8.4	7.4
	14	<u>4.5</u>	<u>7.4</u>	<u>6.2</u>

Average of 2-wk values,  $6.0 \times 10^{-4}$  cc/lb-min

\* Made with FP-555, 5 $\mu$  powder from Brush Beryllium Company.

\*\* Made with chromium-coated powder, R-4630, from Beryllium Corporation.

(CONFIDENTIAL TABLE)

(C) Two additional mixes of Beryllizine-21 were made to evaluate alternative beryllium powders and compare them with Brush Beryllium Company Powder FP-1755, the powder used to make the prototype fuel. Because early work done on the compatibility of hydrazine with KOH-treated beryllium powder indicated that a KOH treatment improved the chemical stability over that of untreated powder, a quantity of FP-1755 powder was treated with KOH and the powder used to make Beryllizine-21. The FP-1755

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigation (cont.)

powder was treated using a solution of 5% KOH in alcohol. The mixture was stirred intermittently and allowed to stand overnight. Then the wet powder was removed and subsequently washed with alcohol, water, and hydrazine. The dielectric constant of the hydrazine wash was 54.5, changed from 46 for the fresh hydrazine. Based on the fact that aluminum and untreated beryllium FP-1755 powders are suitable for gel preparation when the dielectric constant of a hydrazine wash is in the range of 48.5 to 52, the KOH-treated beryllium was determined to be sufficiently free of electrolytes for use in the preparation of gelled fuel. The results of testing Beryllizine-21 made from the KOH-treated powder are listed in Table XXVII and show that no improvement in chemical stability was made to the FP 1755 powder by the KOH treatment.

(C) In order to evaluate in a Beryllizine preparation a beryllium powder with a purity comparable or better than FP-1755 powder, a General Astrometals Corporation powder, GAC 4-88, was tested. This powder caused the dielectric constant of a hydrazine wash to increase to 48, indicating suitability for use in Carbopol-gelled hydrazine. The results of testing Beryllizine made with GAC powder 4-88 are also presented in Table XXVII and show that the powder is comparable to the FP-1755 with regard to chemical stability. The yield stress of 2000 dynes/cm<sup>2</sup> is higher than that for the prototype Beryllizine-21G60 made with FP-1755.

#### (4) Prototype Beryllizine-28

(C) Eleven batches of prototype Beryllizine-28 were made for propellant studies and for ignition and combustion investigations. The components used were the same as those for the prototype Beryllizine-21 preparations. Because of the larger amount of metal used, the Carbopol concentrations were decreased in these batches, and it was concluded that 0.45% was required to give yield stress values in the range from 1400-1600 dynes/cm<sup>2</sup>. The relation of density to temperature over the range from 40° to 120°F was determined as described for Beryllizine-21. The coefficient of thermal expansion was found to be  $3.8 \times 10^{-4} \text{ }^{\circ}\text{F}^{-1}$ .

#### d. Survey of Test Methods

(U) A separate phase of this program had the objective of reviewing heterogeneous propellant properties and test methods and selecting those regarded to be most appropriate to the characterization of the fuels developed during the program. In accord with this objective, the following properties and methods were chosen with the concurrence of the Project Officer:

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XXVII

EVALUATION OF ALTERNATE BERYLLIUM POWDERS IN BERYLLIZINE-21 (C)

<u>Material</u>	<u>Be Powder*</u>	<u>Electrolyte Test</u> <u>N<sub>2</sub>H<sub>4</sub>-Wash.</u>	<u>Yield Stress, dynes/cm<sup>2</sup></u>		<u>Gas Evolution Rate, cc/lb-min x 10<sup>4</sup></u>	
			<u>Initial</u>	<u>1 wk</u>	<u>1 wk</u>	<u>1 mo</u>
		(± 0.5)				
Prototype fuel, Be-21** Typical Mix	FP-1755	48.5	1600	1600	13	10
Be-21** LP-349	KOH-treated FP-1755	54.5	1000	1050	40	Too unstable to store
Be-21** LP-350	GAC 4-88	48.0	2000	1900	15	10

\*FP-1755, powder from The Brush Beryllium Company.

\*\*Gelled with 0.60% Carbopol-940.

## UNCLASSIFIED

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigation (cont.)

<u>Property</u>	<u>Test Instrument</u>	<u>Method</u>
1. Density	Pycnometer	MIL-P-27412 (USAF)
2. Yield Stress	Rising Sphere Rheometer	MIL-P-27412 (USAF)
3. Brookfield-Limiting Flow Characteristic	Brookfield Viscometer	MIL-P-27412 (USAF)
4. High-Shear Viscosity	Ferranti-Shirley Viscometer	Test procedure for Alumizine, Aerojet-General Corporation
5. Characteristic Flow Curve	ASTM D-1092 Viscometer	Test Method ASTM D-1092
6. Smoothness	None (visual inspection)	Newly adopted

(U) While all the above properties are included in the Beryllizine fuel criteria, only four were selected for incorporation into the tentative Beryllizine specification (Appendix C). These are density, yield stress, characteristic flow curve, and smoothness. These four properties, measured in accordance with the specified methods, are sufficient to define the quality of the propellant.

(U) Although density measurements made on aged propellant may be invalid because of bulk growth, density is generally accepted to be essential as a check on the composition of a freshly prepared heterogeneous fuel, if it has been mixed under the vapor pressure of the carrier liquid and it is demonstrated by visual examination that gas bubbles have not been incorporated into the formulation. In view of the qualifications placed on the density of a heterogeneous fuel, it is apparent that the quantitative analysis of the principal fuel components is mandatory. After a fuel having the correct initial density and composition is handled and stored, the density can then be used as an index of the incorporation of gas bubbles or of the mechanical stability of the fuel.

(U) The yield stress referred to in this report is measured by means of the rising sphere rheometer. It is calculated from the force placed on the sphere that is required to strain the gelled fuel to its elastic limit. Because of the complexity of the stress field around the sphere, the relation of the values obtained by this method to the true yield stress is not known. Nevertheless, experience with fuels gelled with polyacrylic acid has shown that rising sphere yield-stress values can be correlated with gel structure as manifested by the ability of the structure to support suspended solids.

## UNCLASSIFIED

Report AFRPL-TR-66-230

### III. A, Chemical Laboratory Investigation (cont.)

Consequently, the yield stress is measured under essentially static conditions; it is inappropriate to relate it to rheological properties, which are measured under dynamic conditions. On the other hand, yield stress and rheological properties such as viscosity must have a common basis related to the structure of the gel. We have shown in our work with Alumizine that there is a high degree of correlation between rising sphere yield stress and high shear viscosity.

(U) Aerojet prefers to use the sphere rather than plates, cylinders, or devices having other geometrical shapes. It has not been demonstrated that these devices provide numbers more closely related to the true yield stress than the values obtained with the sphere. Because a sphere of reasonable size creates force fields of greater magnitude than those caused by plates and cylinders of equal surface area, the rising sphere values can be measured with greater certainty. A more important factor in the choice of the rising sphere method for use in this program is the extensive experience with it by Aerojet, the Rocket Propulsion Laboratory, and other agencies.

(U) Smoothness is a characteristic of heterogeneous fuels that are free of undispersed solid material. This test is made by gross visual examination of specimens smeared across a flat surface by means of a spatula. This is a simple qualitative test that is preferred to the grind-gage test previously used at Aerojet. In the application of the grind gage, undue emphasis was placed on the numbers this device is purported to yield. Experience showed that it could not be used to determine the content of undispersed solids, but that the useful information could be obtained by visual examination of the smoothed surface of the fuel on the test plate. In view of this, the gage was dispensed with, and the smoothness test in its present form was adopted (see section 4.8.7.1 of the tentative Beryllizine specification in Appendix C).

(U) The capillary method is the classical approach to the measurement of viscosity, the ratio of shear stress to shear rate, and the ASTM D-1092 capillary viscometer is a commercially-available instrument that provides shear rate and shear stress data rapidly and economically with relatively small amounts of material. It is practical to obtain about 10 data points in the range of shear rates from 10 to 10,000  $\text{sec}^{-1}$  with 100 ml of fuel and to construct a plot of the data (characteristic flow curve) within a few hours. The characteristic flow curve (Figure 4) presents the flow information in a form most easily used for engineering purposes, making this method preferable to both the plate-cone (Ferranti-Shirley) and rotating-spindle (Brookfield) methods.

(U) The Ferranti-Shirley viscometer permits the measurements of flow behavior up to a shear rate of about 17,300  $\text{sec}^{-1}$ . Although this instrument requires only a few grams of material and plots the shear stress-shear rate data on coordinate paper during the programmed 40-sec run, the

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, A, Chemical Laboratory Investigation (cont.)

results do not provide information significantly more useful than that obtained with the capillary viscometer. Consequently, the cone-plate instrument is not recommended for quality control purposes and is not included in the Beryllizine specification.

(U) The Brookfield limiting-flow characteristic is a relatively crude method of presenting the same rheological information displayed by the characteristic flow curves based on capillary viscometer data. The accuracy and precision of the Brookfield instrument are poor relative to the accuracy and precision of the ASTM D-1092 viscometer. Inasmuch as the greater simplicity and economy of making Brookfield measurements cannot compensate for the inferiority of the data they yield, the capillary method is the preferred one and the Brookfield limiting-flow characteristic is omitted from the Beryllizine specification.

#### e. Quality Control Analyses on Prototype Fuels

(C) To obtain experience with the analytical methods in the Tentative Military Specification for Beryllizine (Appendix C) the batches of prototype propellant have been sampled and tested in the Quality Control Laboratory. Both Beryllizine-21 and -28 were analyzed according to the specification procedures, and the results are presented in Table XXVIII. The reliability of the measurements may be determined by (1) a comparison of the results of analysis with the prepared composition of Beryllizine, and (2) an evaluation of the analyzed composition based on the proximity to 100%. In these respects, the analytical results show good correlation with the theoretically prepared composition. A measure of the uncertainty in the results is shown by the total analyzed concentrations of fuel components having an average deviation of 0.7 from 100%. In the course of the analytical work, it was found that the measurements of the total beryllium concentration were more accurate and precise if a high-purity-standard beryllium\* solution were used to standardize the hydrochloric acid that is the reference reagent in the analytical procedure. This feature of the standardization of the hydrochloric acid is a modification that will be incorporated in the issuance of the tentative specification with the final report.

(U) The effectiveness of the analytical methods for analyzing Beryllizine containing the Cr-coated R-4630 is shown by the results listed in Item 9, Table XXVIII. In the analysis of this Beryllizine no special treatment was required to determine the total beryllium concentration, and, based on the chemistry in the analytical procedure, chromium does not interfere with the analysis for beryllium. This is because only metals forming a fluoride complex would be analyzed as beryllium. The concentration of the chromium in the Cr-coated powder was reported by the manufacturer to be 0.30%, and the prototype fuel containing this powder was not analyzed for the chromium content.

\*Beryllium metal flake, 99.5% Be, spectrographic standard, Catalog No. JM-110, supplied by Harrell-Ash Co., Newtonville, Mass., and described in "Report on Standard Samples and Related Materials for Spectrochemical Analysis," ASTM Pub. No. 58-C, compiled by R. E. Michaelis, National Bureau of Standards, dated 1955.



# CONFIDENTIAL

Report AFRPL-Tr-66-230

TABLE XXVIII

## RESULTS OF CHEMICAL ANALYSES OF PROTOTYPE BERYLLIZINE (U)

Material and Batch Identification	Component Concentrations, %					Totals
	Total Beryllium	Hydrazine*	Carbopol	Water*		
	Ave	Replicates				
Beryllizine-21 (Theoretical)		(21.0) (20.4 based on 97.2% purity of Be powder**)	(78.5)	(0.49)	[0.5]	---
3LP-5 21G52	20.9	21.0 20.7 20.9	78.8	0.38	0.6	100.7
3LP-6 21G48	20.5	20.5 20.5 20.6	78.1	0.54	0.7	99.8
3LP-7 21G48	20.2	20.1 20.2	79.2	0.51	0.9	100.8
3LP-8 21G48	19.7	20.1 19.6 19.5	79.0	0.52	0.9	100.1
3LP-9 21G49	20.2	20.1 20.2	78.4	0.44	0.8	99.8
3LP-10 21G49	20.3	20.1 20.4	78.4	0.49	0.7	99.9
3LP-11 21G49	20.3	20.2 20.3	78.3	0.67	0.9	100.2
3LP-12 21G49	20.5	20.5 20.6 20.5	77.8	0.64	0.6	99.5

\*Hydrazine source analyzed 98.9% of  $N_2H_4$ , 0.6%  $H_2O$  and 0.5% others. The water introduced by hydrazine is shown within brackets.

\*\*The Be powder FP-1755 (Brush Beryllium Co.) was used in all batches except in Item 9 in which was used chromium-coated powder R-4630 (Berylco).

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XXVIII (cont.)

Material and Batch Identification	Component Concentrations, %					Totals
	Total Beryllium	Hydrazine*	Carbopol	Water*		
	Ave	Replicates				
3LP-15 21G49	19.6	19.5 19.6 19.6	77.5	0.65	0.6	98.4
<u>Beryllizine-28</u> (Theoretical)		(28.0) (27.2 based on 97.2% purity of Be powder**)	(71.5)	(0.45)	[0.4]	---
3LP-16 28G45	27.0	27.3 27.3 26.6	72.2	0.48	0.6	100.3
LP-355 28G50	27.5	27.3 27.6 27.5	70.1	0.42	0.6	98.6
LP-356 28G45	26.9	27.1 26.7 27.0	67.5	0.46	0.5	95.4

\*Hydrazine source analyzed 98.9%  $N_2H_4$ , 0.6%  $H_2O$  and 0.5% others. The water introduced by hydrazine is shown within brackets.

\*\*The Be powder FP-1755 (Brush Beryllium Co.) was used in all batches except in Item 9 in which was used chromium-coated powder R-4630 (Berylco).

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, Technical Discussion (cont.)

### B. ENGINEERING INVESTIGATIONS

#### 1. Flow Characterization

(C) The purpose of this portion of the program was to conduct flow studies with Beryllizine to establish the relationship between flow characteristics and the appropriate propellant properties required to design, develop and operate an engine system. Available flow characterization efforts with Alumizine were utilized to the maximum extent possible in order to assess and verify any basic similarities between the two propellants. The rheological properties of the propellant were measured in laboratory equipment to predict the flow behavior of the fuel. Testing was conducted in a flow apparatus which was suitable for verification of the flow behavior of the fuel (as predicted by the laboratory apparatus data). The flow behavior of the fuel in injector elements and manifolds was evaluated to determine pressure drop and any problems of erosion, clogging or separation. Beryllizine containing 21% beryllium powder was used in the tests.

(U) A survey of the state-of-the-art in measurement of gelled-metalized propellant flow was made to determine the most reliable and practical method available. Flowmeter development was not a part of this program.

#### a. Flow-Loop Design and Construction

(U) A flow loop system for the testing of non-Newtonian fluids was designed and constructed early in the program (Figure 7). This system consisted of a fuel feed subsystem, the flow loop proper, and a catch tank subsystem. The fuel feed subsystem, which was also used as a feed system for TCA firings, is described in the section of the Technical Discussion on ignition and combustion. The flow loop was comprised of a test section, associated pressure measuring transducers, and a rigid support on which they were mounted. The test section was fabricated from a 10-ft length of 1/4-in. stainless steel tubing. Taps for pressure measurement were located at 2-ft intervals along the test section to provide the necessary data on pressure drop. The propellant catch tank subsystem, located at the discharge end of the flow loop consisted of a 1-in. flow control valve, a cart-mounted catch tank, and the necessary connecting tubing.

(U) Preliminary to this program a survey was made into the state-of-the-art of measurement of gelled-metalized propellant flow. Conventional flowmeters, weighing systems, and positive displacement systems were the three most popular methods in use. Conventional flowmeters, such as turbine-type, orifices and venturis, were not considered practical because they must be precisely calibrated with the non-Newtonian test media at the exact flow conditions before accurate measurements can be made. Because flow

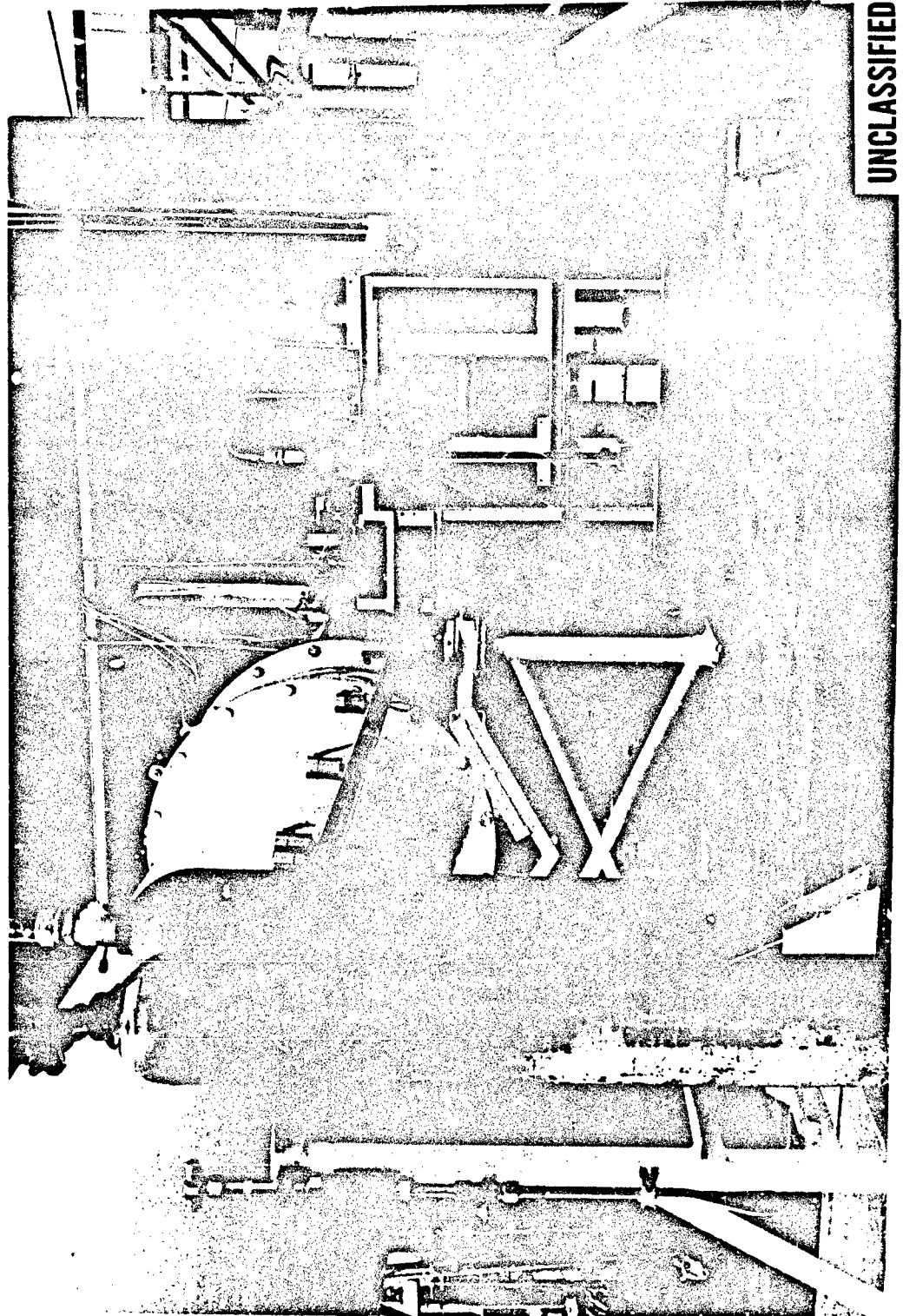


Figure 7. Flow Loop

# UNCLASSIFIED

Report AFRPL-TR-66-230

## III, B, Engineering Investigations (cont.)

data were required in both the laminar and turbulent regions, flowmeter calibrations over the same range would have resulted in a major effort beyond the scope of this program. A weighing system for either the feed or catch tank was not considered suitable because of the mechanical complexity required to eliminate oscillations. Positive displacement of the fuel was selected as the simplest and most practical method of flow measurement. A feed tank containing a butyl rubber bladder was used to contain the Beryllizine which was displaced with pressurized water. The rate of water flow was monitored with a turbine-type flowmeter and the water measurements were assumed to be the volumetric rate of fuel flow. During testing the Beryllizine temperature was monitored with a copper-constantan thermocouple probe and the resultant temperatures were used to obtain the specific gravity of the test fluid. When weight flowrates were required, the specific gravities were converted to propellant densities ( $\text{lb/ft}^3$ ) and combined with the volumetric flowrates ( $\text{ft}^3/\text{sec}$ ) to produce flow-rate data in terms of  $\text{lb/sec}$ . This flow-measuring system was used for both flow and TCA testing.

### b. Data Reduction Techniques

(U) Many attempts have been made to characterize empirically the flow of gelled-metalized fuels but most have not resulted in data in the form that can be directly used by design engineers. The problem of data reduction and presentation was approached early in this program by means of a comprehensive review of the more significant published investigations. A discussion based on this review is presented in the following sections, along with discussions of the methods to be used for the treatment and presentation of test data.

#### (1) General

(U) For a non-Newtonian fluid, the relation between applied shear stress and resultant shear rate is not linear and the ratio of shear stress to shear rate is not a constant as with Newtonian fluids. With a non-Newtonian fluid, the effective viscosity (ratio of shear stress to shear rate) is a function of the shear rate as in equation (1).

$$\tau = \phi (du/dr) \quad (1)$$

$\tau$  = shear stress

$u$  = local velocity

$r$  = radial displacement in flow passage

## UNCLASSIFIED

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

For Newtonian fluids, Poiseville's equation can be applied in the laminar region:

$$\frac{\Delta P}{4L} = \mu \frac{8V}{D} \quad (2)$$

D = diameter of flow passage

$\Delta P$  = pressure drop

L = tube length

V = bulk velocity

$\mu$  = viscosity coefficient

But, in light of equation (1), the functional relation is not linear for a non-Newtonian fluid:

$$\frac{\Delta P}{4L} = \frac{8V}{D} \quad (3)$$

For Newtonian fluids, it can be shown that

$$\frac{du}{dr} = \frac{8V}{D} \quad (4)$$

However, this is not true for non-Newtonian fluids. Thus, data from rotational viscometers, which determine the shear rate  $du/dr$ , cannot be applied directly to a relationship such as equation (2). However, the values for  $\Delta P/4L$  and  $8V/D$  can be determined for non-Newtonian fluids from capillary and pipe flow tests. The ratio of  $\Delta P/4L$  to  $8V/D$  can then be used in the evaluation of parameters of engineering values such as pressure loss and friction factor.

(U) No attempt was made in this program to evaluate the data in terms of some analytical or empirical equation giving the relation between  $\Delta P/4L$  and  $8V/D$ . Instead, the measured  $\Delta P/4L$  versus  $8V/D$  data were used directly to determine the properties for engineering purposes. This approach avoids the difficulties and uncertainties that are involved in attempting to arrive at a general equation relating the flow parameters. However, its drawback is that data must be taken over nearly the full range of operating conditions with only limited extrapolation permitted. Furthermore, the treatment is expressly limited to the single specific fluid undergoing experimental evaluation. No attempt was made to arrive at generalizations that can be used for a number of fluids. Such attempts at generalization have met with little success in the past.

# UNCLASSIFIED

Report AFRPL-TR-66-230

## III, B, Engineering Investigations (cont.)

(U) The approach taken in the flow data analysis was to utilize the measured flow parameters  $\Delta P/4L$  and  $8V/D$  to provide pressure-loss data for design of rocket systems that use the subject propellant.

### (2) Laminar Flow

(U) The presentation of the data in the laminar flow region presents no great difficulty. The relation of equation (3) indicates that a plot of  $\Delta P/4L$  versus  $8V/D$  will yield values that fall on the same curve irrespective of pipe size in laminar flow. This is an established experimental fact.

(U) During the program, the bulk of the laminar flow data was measured with a capillary flow rheometer (ASTM-D-1092). These data, corrected for entrance effects, provide the basic laminar flow curve. This laminar line was used as the basis for validating the design of the full-size tube flow loop used for turbulent flow tests. Since the laminar curve holds true for all tube diameters, the first runs with the full-size tube flow loop were carried out in the laminar region and compared to the curve obtained from the capillary data. The flow loop was considered to be operating properly when the laminar data obtained using it agreed with the laminar data obtained from the capillary apparatus.

(U) There are two phenomena which, if present to any degree in the fluid, would have produced anomalous results in the flow tests. The first of these is time dependency. In general, time dependency manifests itself as an increase (rheopexy) or decrease (thixotropy) in the apparent viscosity of the fluid with time at a constant shear rate. Figure 8 shows the effect of thixotropy (rheoplectic behavior would be just opposite) in capillaries or tubes of various lengths and/or diameters.

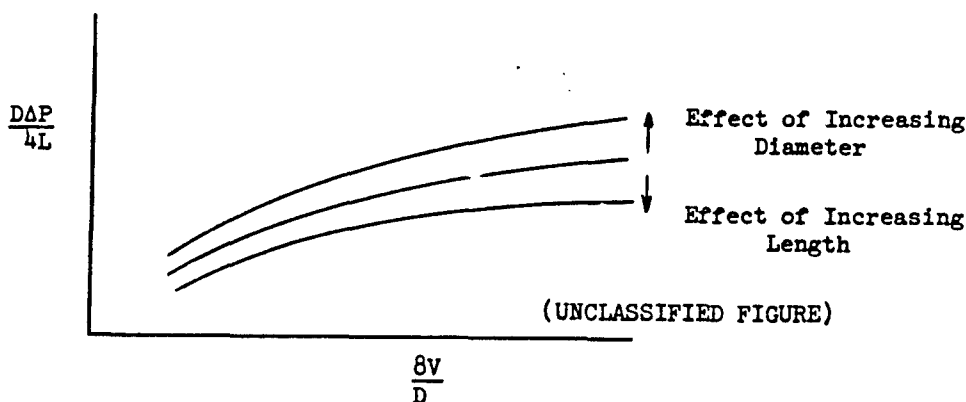


Figure 8. Thixotropic Time Dependency

## UNCLASSIFIED

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

Care was taken in the analysis of the test data to determine the existence of a significant time dependency. All indications at present are that the short flow times typical of capillary and also rocket flow systems preclude the manifestation of a significant time dependency.

(U) The second phenomenon that would have affected the laminar correlation is the wall effect, which produces a "slip velocity" at the tube wall (Reference 2). Figure 9 indicates the effect of wall slip on the flow diagram.

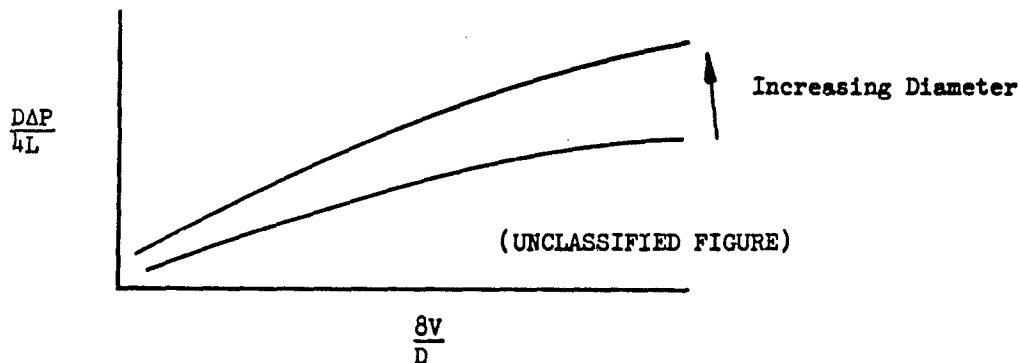


Figure 9. Tube Wall "Slip Velocity" Effect

This appears to have an effect similar to that of thixotropy. However, with slip alone there is no length effect.

(U) The capillary data was corrected for entrance losses by a technique described in Reference 3.

#### (3) Turbulent Flow

(U) Once the laminar flow data are obtained, turbulent flow data are needed to derive a relationship that is valid for the full range of anticipated operating conditions. The combination of laminar and turbulent flow data will provide, in the absence of wall effects and time dependency, all of the information required for presentation of flow parameters and their scale-up to larger pipe sizes.

(U) Figure 10 shows, schematically, a log-log plot of the flow parameters for both the laminar and turbulent flow regions. The turbulent branches of the curve are functions of the tube diameter; this was not the case in laminar flow.



# UNCLASSIFIED

Report AFRPL-TR-66-230

## III, B, Engineering Investigations (cont.)

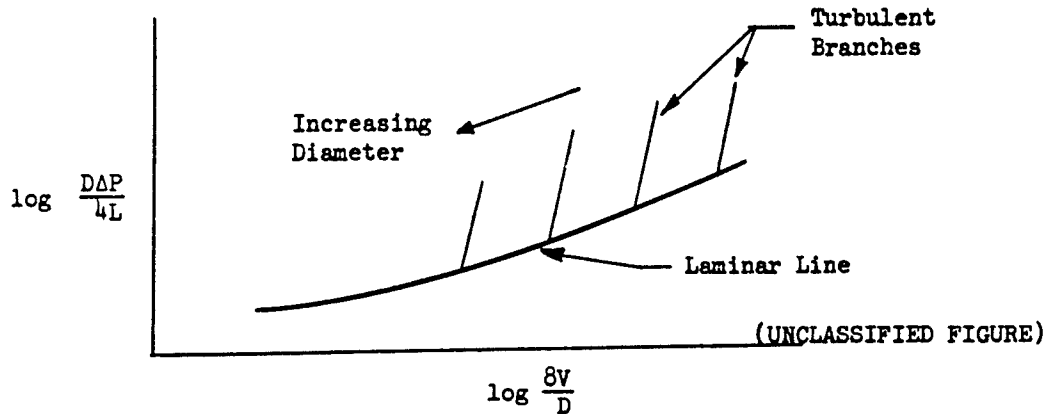


Figure 10. Non-Newtonian Flow Diagram

With Newtonian fluids, the turbulent viscous properties can be correlated (with laminar properties as well) to the Fanning friction factor. The usual correlation is presented by the well known Moody diagram showing friction factor as a function of Reynolds number. By defining a non-Newtonian Reynolds number:

$$R_e' = \frac{DV\rho}{\frac{D\Delta P/8V}{4L D}} \quad (5)$$

$\rho$  = fluid density

$R_e'$  = non-Newtonian Reynolds number

a friction factor - Reynolds number diagram can be produced. With non-Newtonians, although the laminar data plot is one line, the turbulent data are still functions of tube diameter. This method of correlation has met with some success (Reference 4)

(U) Several empirical correlations relating friction factor to Reynolds number have been developed. They are usually effective over the Reynolds number range of 5000 to 100,000 or greater (Reference 4). One convenient form of equation is:

# UNCLASSIFIED

Report AFRPL-TR-66-230

## III, B, Engineering Investigations (cont.)

$$f = \frac{C}{(R_e)^b} \quad (6)$$

$f$  = Fanning friction factor

$C$  = constant

$b$  = exponent (constant)

Since:

$$f = \frac{D \Delta P}{4L} / \rho \frac{V^2}{2g} \quad (7)$$

then from equation (6):

$$\frac{D \Delta P}{2 L V^2} = C(R_e')^{-b} \quad (8)$$

when used with the non-Newtonian Reynolds number ( $R_e'$ ). If the individual terms of the non-Newtonian Reynolds number (Equation 5) are introduced with the apparent viscosity given by equation (2) and are used in the Reynolds number, the following relationship can be derived:

$$\frac{D^{(1+b)} \Delta P}{L} = k V^{(2-b)} = k V^a \quad (9)$$

$k$  = constant which includes apparent viscosity

$a$  = velocity exponent

The exponent,  $a$ , defined in equation (9), is the slope of the turbulent lines plotted on logarithmic coordinates as in Figure 10. Even though a given set of turbulent data may not correlate properly on a Moody diagram, by referring to equation (9), we note that a logarithmic plot of  $D^{(1+b)} \Delta P / L$  versus  $V$  should give a straight line. The value of  $b$  is the slope of the turbulent line on a Moody diagram, or the negative of  $(2-a)$ . The slope of all of the turbulent branches in a plot such as Figure 10 may not be the same, but usually they are sufficiently close together to allow an effective average to be taken. Figure 11 shows the plot of  $D^{1+b} \Delta P / 4L$  versus  $V$ .

# UNCLASSIFIED

Report AFRPL-TR-66-230

## III,B, Engineering Investigations (cont.)

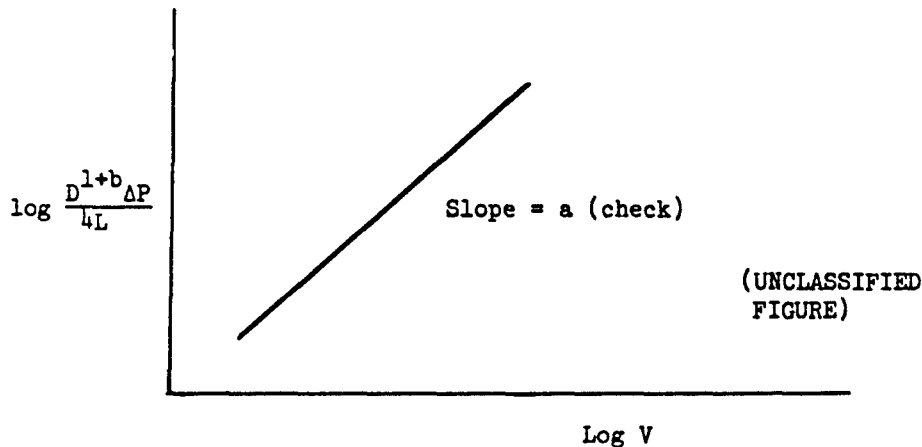


Figure 11. Adjusted Shearing Stress and Turbulent Velocity Relationship

If this correlation proves successful, then various diameters of pipe can be inserted to obtain  $\Delta P/4L$  and  $8V/D$  using Figure 11. These values can be included on the  $\Delta P/4L$  versus  $8V/D$  plot as in Figure 10. In practice at least three tube diameters should be used to obtain data for Figure 10 but once the correlation is achieved, then values for any diameter can be obtained to construct Figure 10 to very large diameter.

(U) Furthermore, once the extrapolation to scaled-up pipe diameter is made on Figure 10, the Reynolds numbers at which the transition to turbulence occurs can be determined for any pipe size by locating the intersection of the straight turbulent lines with the laminar line. The Reynolds number (equation 5) at each intersection is the critical Reynolds number. Some authors have indicated that the critical Reynolds number for a given fluid is a constant at all diameters (Reference 4), but this has been shown to be incorrect (Reference 3).

(U) The above correlation assumes that the non-Newtonian Reynolds number remains essentially constant. For certain pseudoplastic fluids, however, this is not the case. Since many pseudoplastic fluids obey the power-law:

$$\frac{\Delta P}{4L} = K \left( \frac{8V}{D} \right)^n \quad (10)$$

## UNCLASSIFIED

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

where  $n$  is the slope of the logarithmic plot of  $D\Delta P/4L$  versus  $8V/D$ . By rearrangement of equation (6) the exponent of velocity is given by:

$$a = (2 - 2b + b n) \quad (11)$$

which will allow evaluation of  $b$  when the slope of the turbulent lines,  $a$ , is known along with  $n$ . For the ideal power-law fluid,  $n$  is a constant.

(U) The  $D^{(1+b)}\Delta P/4L$  versus  $V$  plot can again be constructed, as in Figure 12, to produce the correlation for the pseudoplastic.

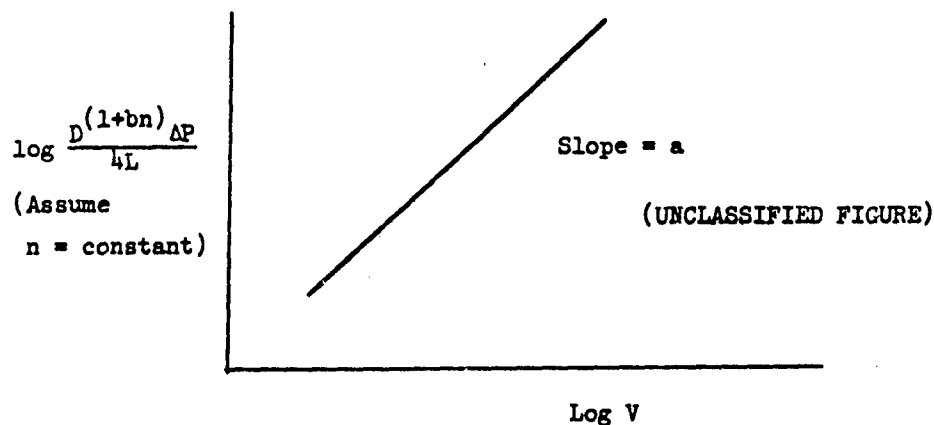


Figure 12. Correlation for Pseudoplastic Flow

(U) There may be a transition flow region with pseudoplastics that is usually not present in other non-Newtonians. This transition usually manifests itself as an abrupt change of slope in the turbulent branch lines. A typical turbulent transition on log-log coordinates is shown in Figure 13.

# UNCLASSIFIED

Report AFRPL-TR-66-230

## III, B, Engineering Investigations (cont.)

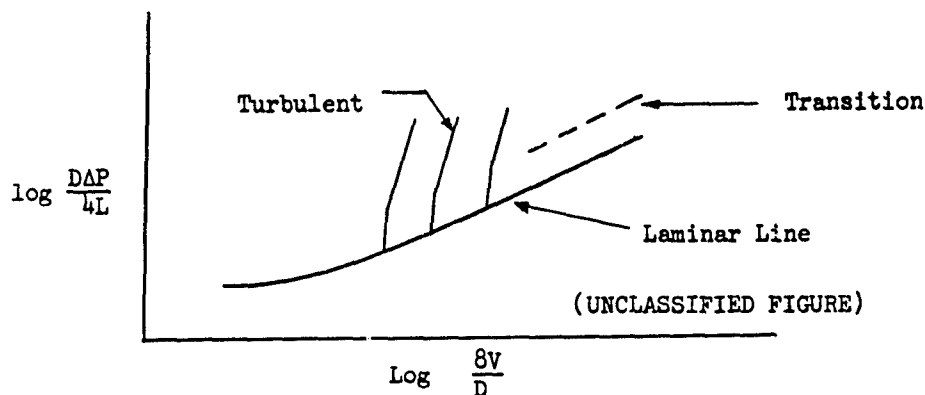


Figure 13. Adjusted Shearing Stress and Turbulent Velocity Relationship with Transition Region

Because the transition regions are plotted as straight lines, the same treatment as outlined above can be applied for scale-up of the flow diameter and for obtaining critical Reynolds number.

(U) With anomalous fluids, the exponent  $b$  may be difficult to determine for use in the above techniques, but another method is still available. Equation (9) can be arranged with the exponent  $(1 + b)$  equal to some general unknown  $X$ . Then, rearranging:

$$\frac{\Delta P}{LV^a} = \frac{k}{D^X} \quad (12)$$

The exponent  $a$  is still the slope of the turbulent branches of  $DAP/4L$  versus  $8V/D$  on log-log coordinates. To determine the exponent of diameter,  $x$ , a log-log plot of  $\Delta P/LV^a$  versus  $D$  is made from the turbulent data. At least three tube diameters are required. The slope of the line is equal to  $X$  (see Figure 14).

# CONFIDENTIAL

Report AFRPL-TR-66-230

## III, B, Engineering Investigations (cont.)

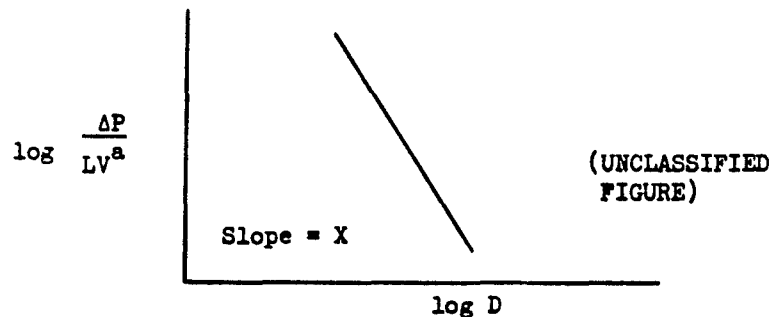


Figure 14. Flow Diameter Exponent in the Turbulent Region for Anomalous Fluids

With this information, a log-log plot of  $D^X \Delta P / 4L$  versus  $V$  can be constructed. This curve is similar to Figure 11 and can be used directly for turbulent scale-up.

(U) Where a fluid is anomalous as regards the friction factor-Reynolds number plot or power-law, as noted above, but behaves like the fluid of Figure 12, an alternative method for obtaining a correlation is available. With this method, the turbulent data for at least three diameters are taken and plotted parallel with the best average slope for the three diameters (Figure 15).

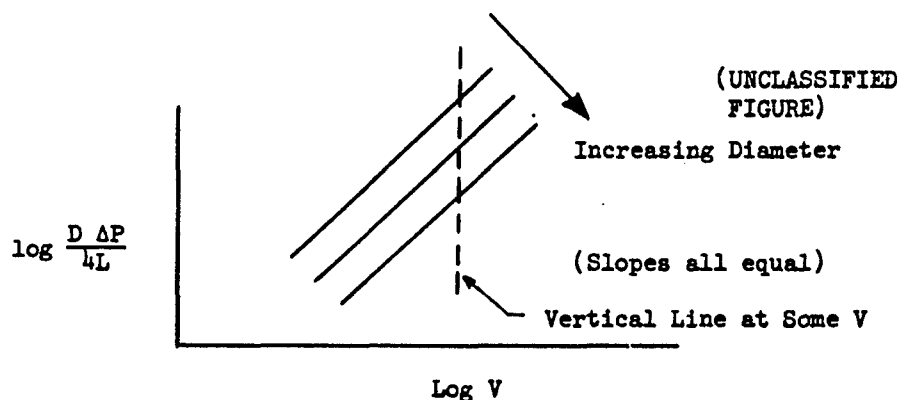


Figure 15. Alternative Correlation for Anomalous Fluids

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

A vertical line is then drawn through all three lines at some arbitrary velocity (Figure 15). The values of  $D\Delta P/4L$  at each intersection of the vertical line with the turbulent lines are plotted versus the diameters. The slope of this line gives the exponent,  $x$ , of the diameter as in Figure 14. Again, the log-log plot of  $D^x\Delta P/4L$  versus  $V$  can be made as in Figure 11, and the scale-up carried out.

(U) By use of any one of the methods outlined above, the plot of  $D\Delta P/4L$  versus  $8V/D$  can be obtained.

(U) The specific behavior of the fluid determines which method can be applied. A useful engineering curve can be produced with this plot, giving pressure loss for some given pipe length (e.g., per 100 ft of pipe) versus the volumetric flow rate (Figure 16).

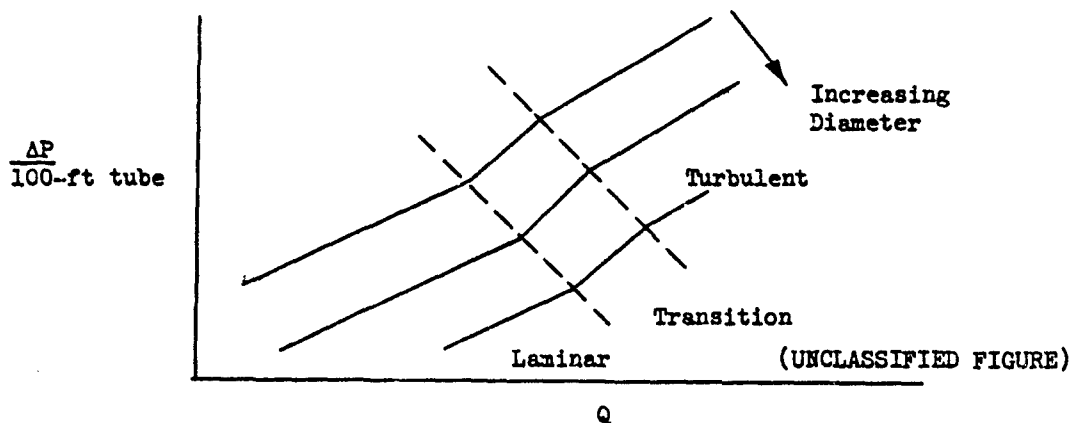


Figure 16. Method of Tube Flow Data Presentation

#### c. Test Program

##### (1) Testing with the Capillary Viscometer

(C) The success of a flow test program with a gelled-metalized propellant depends largely upon the reproducibility of yield stress, within a reasonable tolerance, from batch to batch of the heterogeneous fluid. This requirement is necessary because there is no known method of

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigation (cont.)

correcting pressure losses for propellants having a wide range of yield stresses. The first test series was conducted in the Chemistry Laboratory with the ASTM-D-1092 capillary viscometer to characterize Beryllizine-21 in the laminar regime. The temperatures of the test fluid and the capillary viscometer were held constant at 77°F. Tests were performed from a shearing rate ( $8V/D$ ) of approximately  $10 \text{ sec}^{-1}$  to  $10^4 \text{ sec}^{-1}$ . The data are presented in Figure 4 as a plot of shearing rate ( $8V/D$ ) versus shearing stress ( $DAP/4L$ ), for the two batches of Beryllizine-21 tested. Also shown are the results of tests with Alumizine-43G conducted over the same range of shearing rates. The plotted data indicate that the flow characteristics of Beryllizine-21 are the same as those of Alumizine-43G in the laminar regime since the slopes of the curves are nearly identical. Additional evidence that the fluids behave in similar fashion is the fact that the curve corresponding to the material of highest yield stress is uppermost and the curves representing the fluids of lesser yield stress extend progressively downward. The values of yield stress were obtained with Aerojet's rising sphere rheometer which is discussed in the chemistry section of this report.

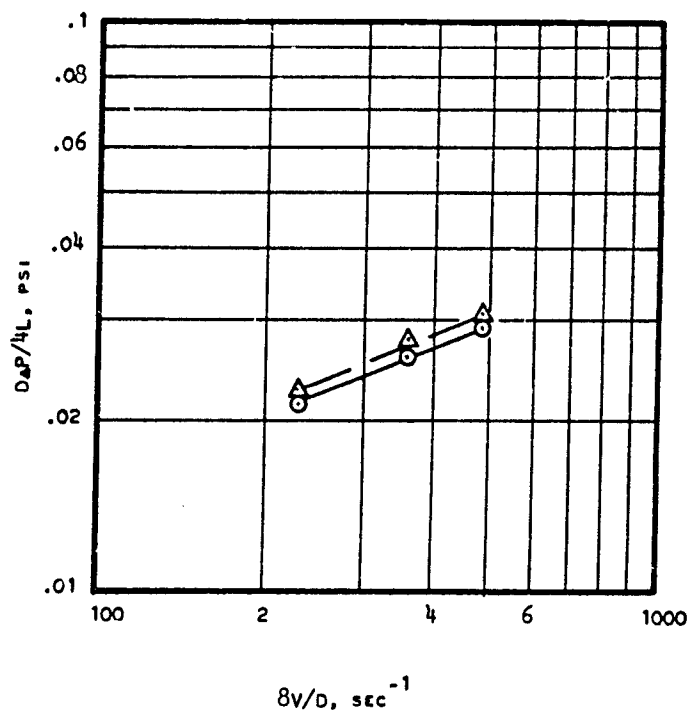
(C) Capillary rheometers are very effective in obtaining viscous flow data for non-Newtonian fluids. However, certain corrections should be made to the data thus measured. Reference 3 discusses the Hagenback-Couette entrance correction. This correction was applied to the data obtained from the capillary rheometer. The extent of this correction is shown in Figure 17. The corrected pressure gradient is obtained by taking flow measurements in two capillaries having the same diameter but different lengths. Figure 17 indicates that the correction is rather small, as is expected from the theory. Actually, the comparison is made to only one of the capillaries (the longer). Although the two different lengths should give the same line on the curve, the data given here for the 2.399-in. long and the 11.993-in. long tube (diameter 0.0625 in.) showed slightly different lines with the longer-tube data being below the shorter-tube data (Figure 18). This phenomenon is indicative of a slight thixotropic effect. However, since the curve indicates that the effect is rather small (probably well within the data scatter for the flow-loop) at high flow rates, and because a convenient correlation is, at present, impractical if thixotropy is considered, this phenomenon was ignored in data correlations.

(U) In the laminar-flow region, the shear diagram ( $D P/4L$  versus  $8V/D$ ) is not a function of the flow-passage diameter. Thus the laminar-flow data taken from straight-tube tests should fall on the same line as those taken for the more carefully controlled capillary tests. Since this comparison was reasonably close, it can be assumed that the straight-tube flow apparatus was sufficiently well designed to give reliable data.



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Report AFRPL-TR-66-230



Hagenback-Couette Entrance  
Correction for Beryllizine-21  
ASTM D-1092 Capillary Extrusion  
Rheometer

○ Measured Data

△ Corrected for Entrance Effects

Yield Stress = 1400 dynes/cm<sup>2</sup>

Temp = 77°F

(Measured and corrected curves are for  
the 11.99-in. long capillary)

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Figure 17. Entrance Correction for Capillary Viscometer (U)

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Report AFRPL-TR-66-230

### III, B, Engineering Investigation (cont.)

#### (2) Testing with the Flow Loop System

##### (a) Water-Calibration Tests

(U) After completion of the flow loop, it was calibrated first with distilled water. Five groups of tests were conducted for a total of 47 tests. The rate of flow ranged from 0.0996 lb/sec to 1.000 lb/sec. The corresponding Reynolds numbers were  $1.33 \times 10^4$  to  $1.33 \times 10^5$ . The data points which had less than 3% instrumentation error are plotted in Figure 19, and are compared with the theoretical predictions. The close agreement of the experimental values with those calculated from the Crane Handbook show that the flow loop was operating properly.

##### (b) Beryllizine-Calibration Tests

(C) Two groups of tests were performed for a total of 16 tests. The rate of flow ranged from 0.0804 to 0.565 lb/sec. This range was chosen to stay within the laminar region of flow. The average yield stress of the first group of tests (1K-2A-106) was 950 dynes/cm<sup>2</sup> and the second group (1K-2A-107) was 850 dynes/cm<sup>2</sup>. The data from the tests are tabulated in Table XXIX.

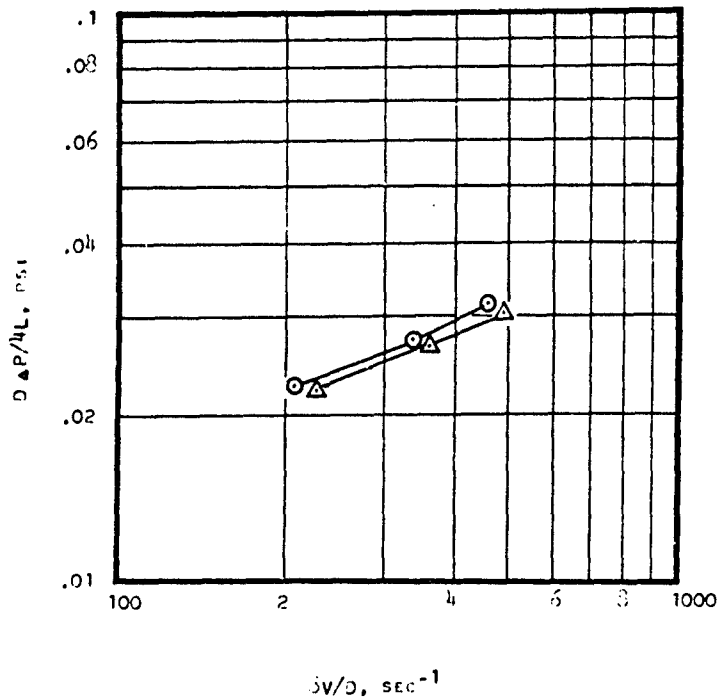
##### (c) Tests in the Laminar Region of Flow

(U) Unfortunately, because of lack of temperature control facilities the properties of Beryllizine used in the flow-loop tests were not identical to those of the Beryllizine used in the capillary tests. Not only did the basic gel consistency vary (that is, the nominal yield stress value) between batches used, but the temperature in some of the flow-loop tests varied from the nominal 77°F at which gel yield stress is evaluated. However, this does not preclude an effective comparison of the capillary data with the flow loop data.

(U) If the gel properties are assumed to be reasonably constant and only the value of the yield stress varies, then the lines on the shear diagram should be parallel with the gels having higher yield stress above those having lower yield stress. The capillary flow gel sample used in the tests had a yield stress of 1400 dynes/cm<sup>2</sup>. The two batches run in the 1/4-in. flow loop had yield stress values of 1550 and 850 dynes/cm<sup>2</sup>. If the flow loop is, in fact, operating properly with no spurious flow effects present, the capillary data should fall between the flow loop data. Figure 20 shows that a linear extrapolation of the capillary data falls between the tube flow data and somewhat closer to the line for the gel of higher yield strength. This is very close to what would be expected. Thus, on the basis of the available data, it appears that the flow loop was operating properly, and the two sets of data are comparable.

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Report AFRPL-TR-66-230



Indication of Thixotropy in Beryllizine-21  
ASTM D-1092 Capillary Extrusion Rheometer

Yield Stress of Beryllizine-21 = 1400 dynes/cm<sup>3</sup>

Capillary Average Diameter = 0.0625 in.

Capillary Length:

○ 2.399 in.

Δ 11.993 in.

Temp = 77°F

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Figure 18. Beryllizine-21 Thixotropy (U)

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Report AFRPL-TR-66-230

TABLE XXIX

TEST DATA FROM FLOW LOOP  
CALIBRATIONS WITH BERYLLIZINE (u)

<u>Test No.</u>	<u>Flow Rate, lb/sec</u>	<u>Instr. Error, %</u>	<u>ΔP/ft, psi/ft</u>	<u>Instr. Error, %</u>
106-1 *	Test to bleed air from test section.			
-2	Test to bleed air from test section.			
-3	0.1057	$\pm 1.0$	19.15	0.71
-4	0.0829	$\pm 1.0$	16.75	0.81
-5	0.0829	$\pm 1.0$	17.32	0.78
-6	No Beryllizine flow.			
-7	0.1002	$\pm 1.0$	18.65	0.72
-8	0.0984	$\pm 1.0$	18.53	0.73
107-1 **	Test to bleed air from test section.			
-2	0.565	$\pm 0.5$	26.6	0.568
-3	0.473	$\pm 0.5$	30.19	0.50
-4	0.267	$\pm 0.5$	24.83	0.609
-5	0.151	$\pm 0.5$	18.64	0.81
-6	0.0804	$\pm 0.5$	13.96	1.08
-7	0.252	$\pm 0.5$	23.99	0.629
-8	0.419			

\* Average Beryllizine yield stress was 950 dynes-cm<sup>-2</sup>.

\*\* Average Beryllizine yield stress was 850 dynes-cm<sup>-2</sup>.

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Report AFRPL-TR-66-230

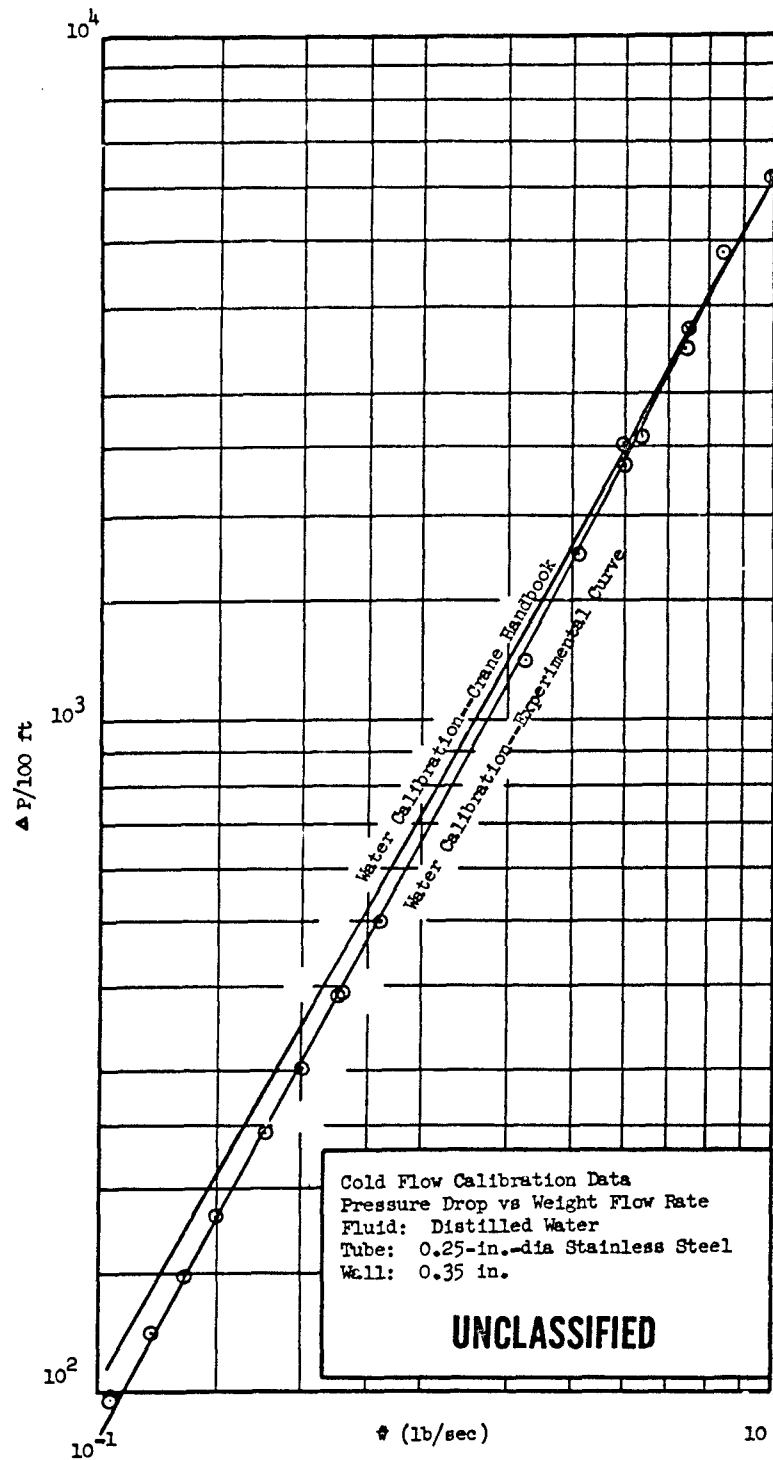


Figure 19. Test Data, Water Flow

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Report AFRPL-TR-66-230

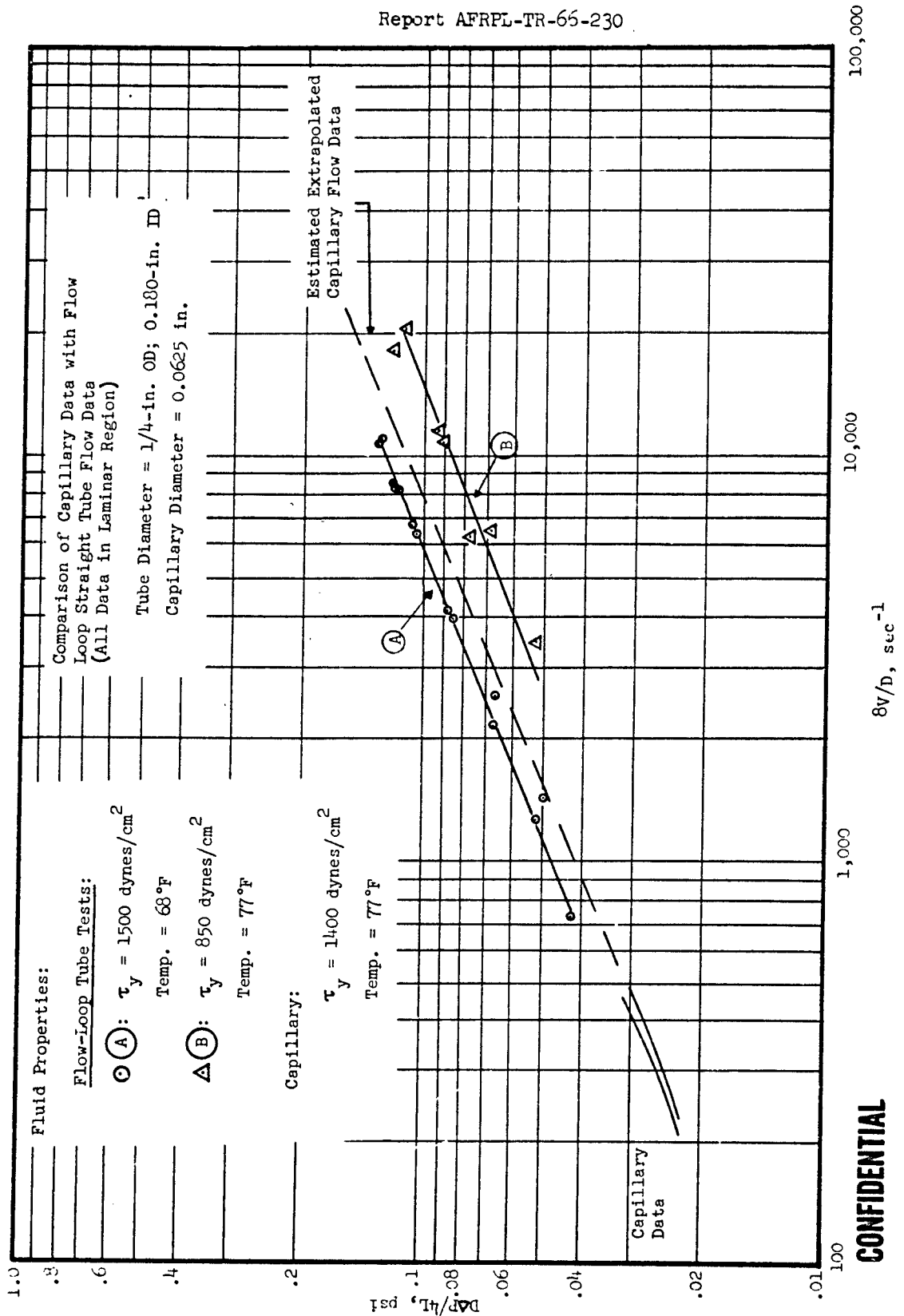


Figure 20. Comparison of Flow Data for Beryllizine-21 (U)

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Report AFRPL-TR-66-230

### III, B, Engineering Investigation (cont.)

(U) Unless some shear equation can be obtained (e.g., modified power law), there can be no really effective correlation of the flow loop data for a more exact comparison to the capillary data. This further bears out the need for careful temperature and batch control.

#### (d) Tests in the Turbulent Region of Flow

(C) When the tests in the laminar region of flow had been completed and it was assured that the flow loop was operating properly, the Beryllizine-21 was tested in the turbulent regime. The objective of these tests was to determine if Beryllizine has the same flow characteristics as Alumizine in the turbulent region of flow. Tests were performed from a shearing rate of  $4.02 \times 10^4 \text{ sec}^{-1}$  to  $5.98 \times 10^4 \text{ sec}^{-1}$ . The Beryllizine temperature during the tests was  $68.6^\circ\text{F}$ . The data may be found in Table XXX and are presented in Figure 21 as a plot of shearing rate ( $8V/D$ ) versus shearing stress ( $D\Delta P/4L$ ) for the batch of Beryllizine-21 tested. Also shown are the results of tests with Alumizine in the turbulent regime. The similarity of the plotted data indicate that the flow characteristics of the fluids are the same in the turbulent region.

(C) Figure 22 is a comparison of characteristic flow curves obtained with Alumizine (References 5 and 6) and Beryllizine. The laminar flow line for the Beryllizine was graphically extrapolated from capillary data obtained with Beryllizine samples taken from the batch used for the flow loop tests. The turbulent branch was constructed from the flow loop test data. Unfortunately, the properties of the Alumizine are unknown (i.e., yield stress and specific gravity) so a direct numerical comparison of flow characteristics in the form of effective Reynolds numbers is impossible. However, the transitional shearing stress of the Alumizine corresponding to a 0.18-in. dia tube was obtained graphically and compared to the value measured with Beryllizine at the same diameter. The transitional points of both fluids were found to occur at nearly identical shearing rates which further indicates similarity in flow characteristics.

#### (e) Injector Flow Testing

(U) The injectors built for the ignition and combustion portion of the program were used for evaluating the flow behavior of the fuel. Of particular interest were the flow characteristics in injectors in terms of pressure drop and flowrate. Also desired were observations of any problems of erosion, clogging, or fuel separation.

(C) During the design phase it was assumed that Beryllizine would behave like Alumizine-43 when flowing through an injector. Figure 23 shows some results obtained from the Improved Titan

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Report AFRPL-TR-66-230

TABLE XXX

TEST DATA FROM FLOW LOOP TESTING  
IN THE TURBULENT REGIME (u)

<u>Test No.</u>	<u>W, lb/sec</u>	<u>ΔP/L, psi/ft</u>	<u>Shearing Rate, 8V/D, sec<sup>-1</sup></u>	<u>Shearing Stress, ΔP/4L, psi</u>
110-1 *	1.379	145.4	$5.98 \times 10^4$	$5.46 \times 10^{-1}$
-2	1.379	143.3	$5.98 \times 10^4$	$5.38 \times 10^{-1}$
-3	1.050	88.7	$4.57 \times 10^4$	$3.32 \times 10^{-1}$
-4	1.056	84.8	$4.60 \times 10^4$	$3.18 \times 10^{-1}$
-5	0.924	69.7	$4.02 \times 10^4$	$2.61 \times 10^{-1}$

<u>Test Fluid</u>	<u>Properties</u>	<u>Dia. of Flow Tube, in.</u>
Beryllizine-21	Yield Stress = $1300 \frac{\text{dynes}}{\text{cm}^2}$ **	0.18
	Specific Gravity = $1.114 \text{ gm-cc}^{-1}$ **	

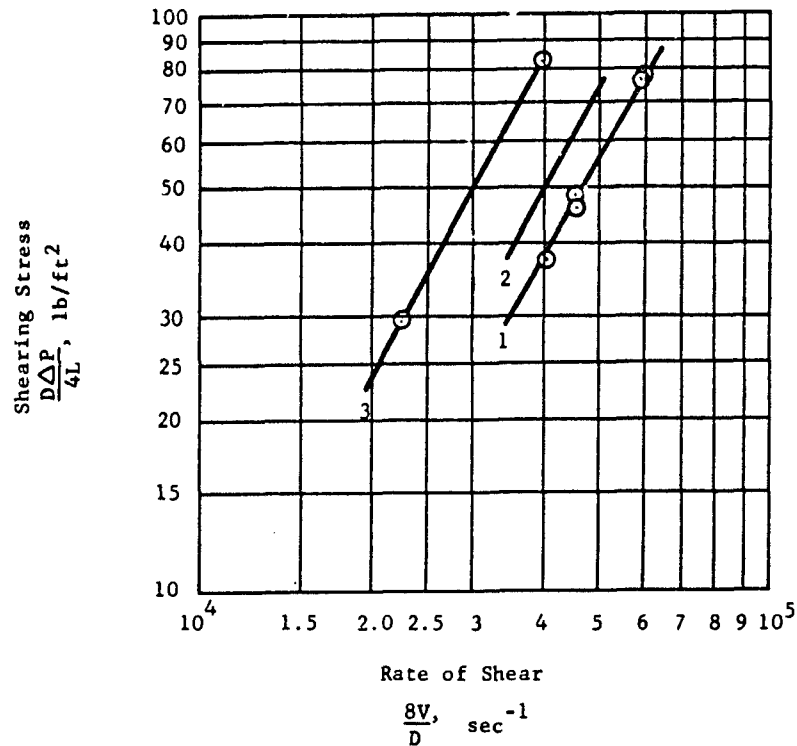
\* Beryllizine temperature was 68.6°F during all tests.

\*\* Measurements were taken at 77°F.



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Report AFRPL-TR-66-230



Curve No.	Test Fluid	Properties	Dia. of Flow Tube, in.	Source of Data
1	Beryllizine-21	Yield Stress = 1300 $\frac{\text{dynes}}{\text{cm}^2}$ at 77°F Specific Grav. = 1.114 $\text{gms/cm}^3$ at 77°F	0.18	AGC flow tests, HEMP Program, AF 04(611)-10783
2	Alumizine-43G	Unknown	0.18	Reference 5
3	Alumizine-43G	Apparent Viscosity = 27,000 cp at 6 RPM (Brookfield)	0.277	Reference 6

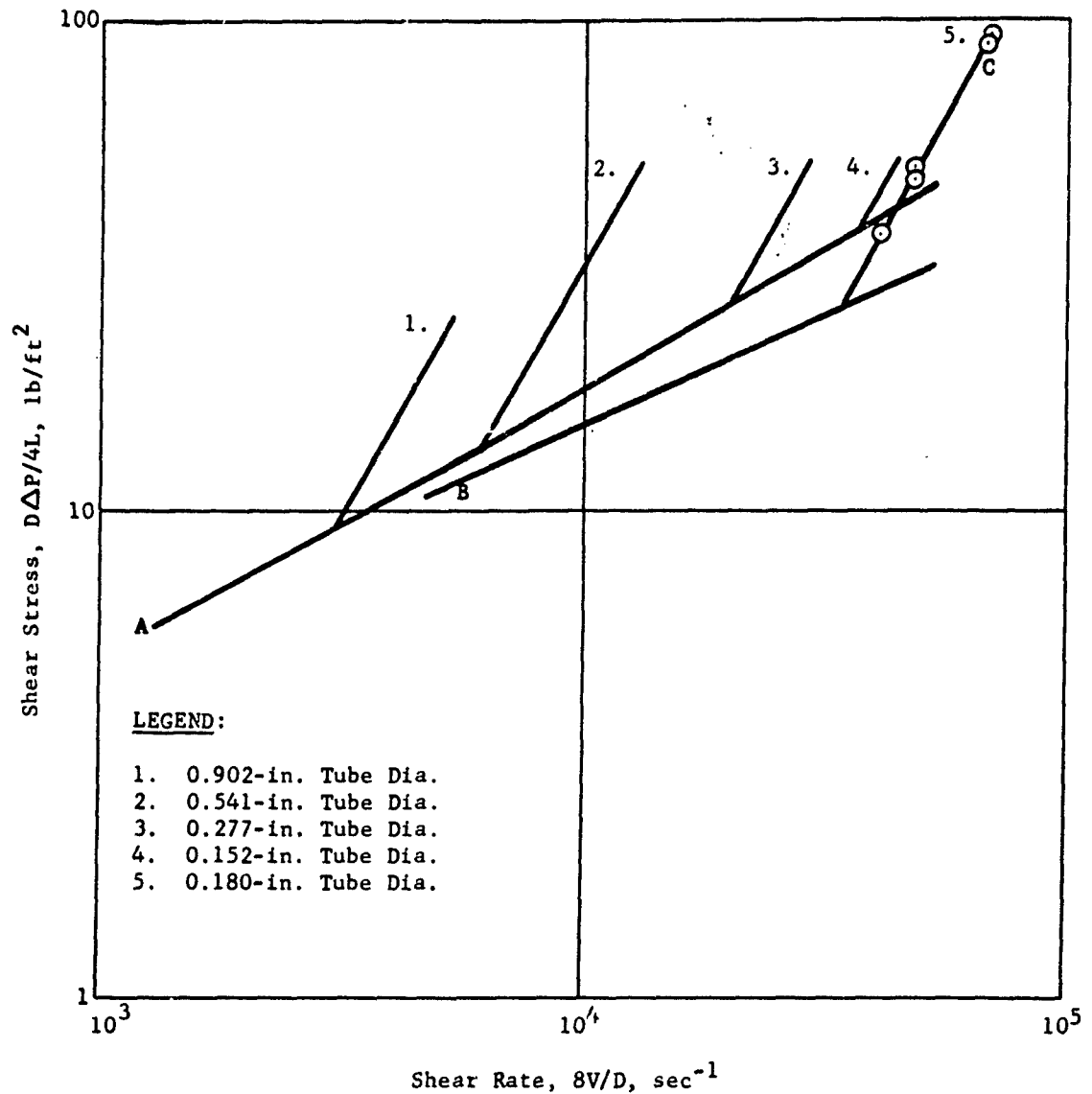
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Figure 21. Characteristic Flow Curve in the Turbulent Regime for Alumizine-43 and Beryllizine-21 (U)

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Report AFRPL-TR-66-230



Curve	Test Fluid	Properties at 77°F	Source of Data
A	Alumizine-43G	Unknown	Reference 6
B	Beryllizine-21	Yield Stress = 1300 dynes-cm <sup>2</sup> Specific Grav. = 1.114 gm-cc <sup>-1</sup>	ASTM-D-1092 Viscometer
C	Beryllizine-21	Yield Stress = 1300 dynes-cm <sup>2</sup> Specific Grav. = 1.114 gm-cc <sup>-1</sup>	Flow Loop, 1/4-in. Test Section

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Figure 22. Characteristic Flow Curve for Alumizine and Beryllizine-21 (U)

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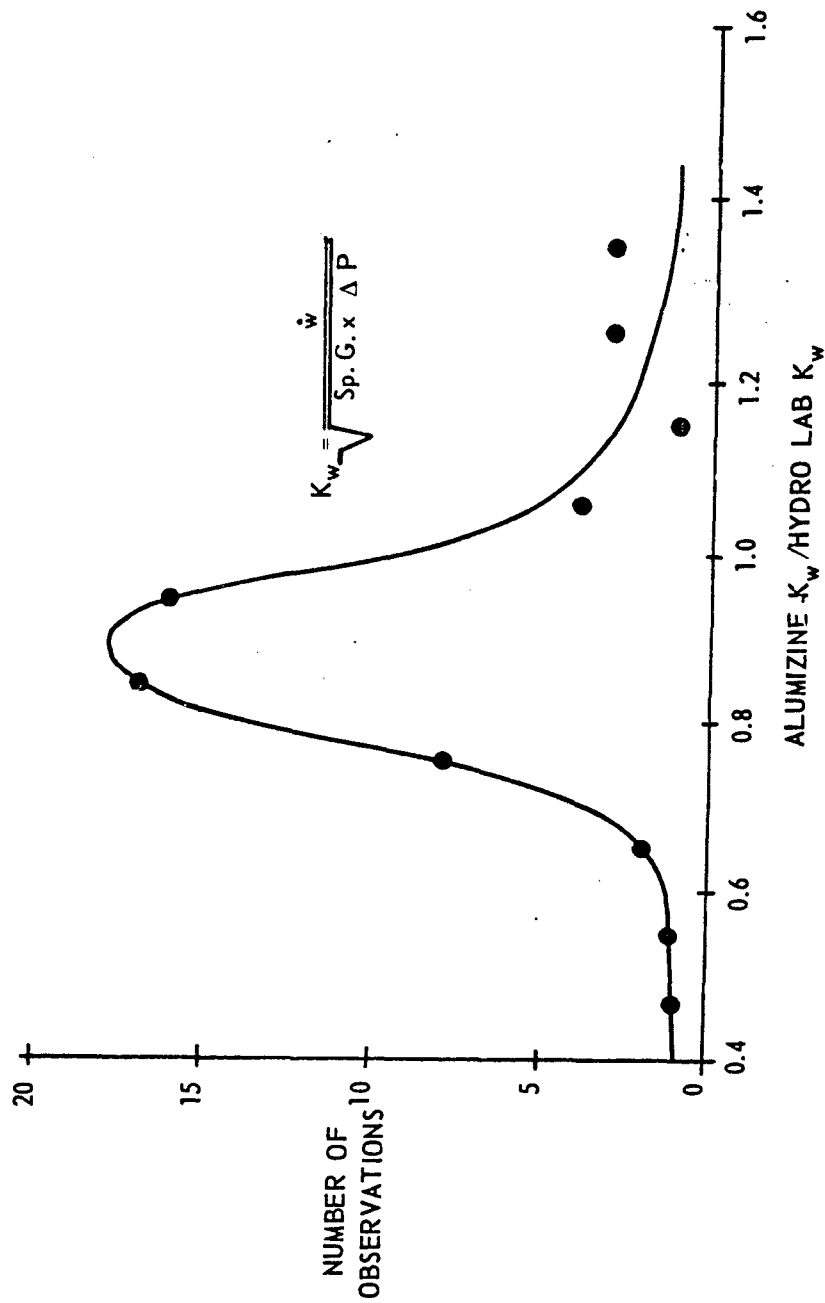


Figure 23. Comparison of Alumazine-43  $K_w$  with Water  $K_w$

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Report AFRPL-TR-66-230

### III, B, Engineering Investigation (cont.)

(Titan II-A) Predevelopment Program with regard to observations of the difference between the flows of water and Alumizine-43 through injectors. It should be pointed out that the maximum number of observations resulted in a hydraulic factor for Alumizine-43 that was approximately 85% of that obtained for water. Because no other data were available, it was assumed that the hydraulic factor for Beryllizine would be 85% of that calculated for water for engineering design purposes. The data for the injectors, presented in Tables XXXI and XXXII, show that the hydraulic efficiency used during the design phase was correct and the flow characteristics through an injector are similar for Alumizine-43 and Beryllizine-21.

(U) The injectors were carefully inspected after each test for indications of erosion caused by Beryllizine flow. The internal surfaces of neither the small fuel orifices (0.048- to 0.063-in. dia in the two injectors), where the highest velocity occurred, nor the fuel manifolds, where the lowest velocity occurred, showed any signs of erosion. After each test the internal surfaces of the fuel orifices were inspected and found coated with a film of Beryllizine. The posttest purges with nitrogen gas successfully emptied most of the Beryllizine from the injectors, but left fuel-coated surfaces and deposits of Beryllizine in the internal cavities and corners. The residual fuel was easily removed with hot water and detergent. The orifices were inspected for signs of clogging before and after flushing with hot water. Neither the test data nor observations of the test hardware after the tests indicated signs of clogging. In all cases, unseparated deposits of Beryllizine were found in the injector; there is no evidence, based upon visual observations, of liquid and solid phase separation of the fuel in the injectors.

#### 2. Ignition and Combustion Technology

(C) This portion of the program was concerned with the development of techniques for the ignition and efficient combustion of Beryllizine at near-optimum mixture ratios, using nitrogen tetroxide and 98% hydrogen peroxide as the oxidizers. The investigations were conducted with uncooled thrust chamber assemblies operating at nominal levels of thrust and chamber pressure of 1000 lb and 1000 psia, respectively. The initial tests were conducted with the  $N_2O_4$ /Beryllizine-21 system. The metal content of this fuel was established to produce the highest value of theoretical specific impulse with  $N_2O_4$ . The balance of the test program was conducted with 98%  $H_2O_2$  and Beryllizine-28, the metal content of which had been optimized for maximum performance of this propellant combination. Applicable technology

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Report AFRPL-TR-66-230

TABLE XXXI

## HYDRAULIC PERFORMANCE, MODIFIED CENTROJET INJECTOR (u)

### HOT-CORE SECTION, FUEL CIRCUIT

### DESIGN VALUES

<u>Flow Rate, lb/sec</u>		<u>Pressure</u>		<u>Kv</u>	
<u>Water</u>	<u>Fuel</u>	<u>Drop, psi</u>	<u>C<sub>d</sub></u>	<u>Water</u>	<u>Fuel</u>
0.309	0.276	108.6	0.6	0.029	0.025

## VALUES FROM WATER TESTING

<u>Flow Rate,</u> <u>Water, lb/sec</u>	<u>Pressure</u> <u>Drop, psi</u>	<u>C<sub>d</sub></u>	<u>K<sub>w</sub>,</u> <u>Water</u>
0.309	93.4	0.73	0.032

## VALUES FROM TEST FIRINGS WITH BERYLLIZINE-21

<u>Test No.</u>	<u>Flow Rate Fuel, lb/sec</u>	<u>Pressure Drop, psi</u>	<u>C<sub>d</sub></u>	<u>K<sub>w</sub></u>	<u>Average K<sub>w</sub></u>
101	0.266	108.1	0.56	0.024	0.024
102	0.267	108.9	0.56	0.024	
103	0.268	112.6	0.56	0.024	

## Kw COMPARISONS

$$Kw \text{ (Fuel-Avg)}/Kw \text{ (Design Value, Water)} = 83\% \text{ (85\% was assumed)}$$
$$Kw \text{ (Fuel-Avg)}/Kw \text{ (Design Value, Fuel)} = 96\% \text{ (100\% was goal)}$$
$$K_w (\text{Fuel-Avg}) / K_w (\text{Test Value, Water}) = 75\% \text{ (85\% was goal, error was caused by decreasing } C_d)$$

where  $K_w = \dot{W} / \sqrt{S.G. (\Delta P)}$

$\dot{W}$  = flow rate of test fluid, lb/sec  
S.G. = specific gravity of test fluid  
 $\Delta P$  = resultant pressure loss, psi

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Report AFRPL-TR-66-230

### TABLE XXXII

### HYDRAULIC PERFORMANCE, MODIFIED CENTROJET INJECTOR (u)

### MAIN SECTION, FUEL CIRCUIT

## DESIGN VALUES

<u>Flow Rate, lb/sec</u>		<u>Pressure Drop, psi</u>	<u>C<sub>d</sub></u>	<u>K<sub>v</sub></u>	
<u>Water</u>	<u>Fuel</u>			<u>Water</u>	<u>Fuel</u>
2.07	1.844	99.3	0.85	0.208	0.177

### VALUES FROM WATER TESTING

<u>Flow Rate,</u> <u>Water, lb/sec</u>	<u>Pressure</u> <u>Drop, psi</u>	<u>C<sub>d</sub></u>	<u>K<sub>v</sub>,</u> <u>Water</u>
2.07	121	0.77	0.188

VALUES FROM TEST FIRINGS WITH BERYLLIZINE-21

<u>Test No.</u>	<u>Flow Rate Fuel, lb/sec</u>	<u>Pressure Drop, psi</u>	<u>C<sub>d</sub></u>	<u>K<sub>v</sub></u>	<u>Average K<sub>v</sub></u>
101	1.754	100.3	0.91	0.166	0.161
102	1.740	92.9	0.94	0.159	
103	1.764	105.5	0.95	0.158	

## Kw COMPARISONS

$$K_w (\text{Beryllizine-21-Avg}) / K_w (\text{Design Value, Water}) = 77.5\% \text{ (85\% was assumed, error was caused by } C_d \text{)}$$

$$Kw \text{ (Beryllizine-21-Avg)} / Kw(\text{Design Value, Fuel}) = 91\% \text{ (100\% was goal)}$$

$$K_w (\text{Beryllizine-21-Avg}) / K_w (\text{Test Value, Water}) = 85.5\% \text{ (85\% was goal)}$$

where  $K_v = \dot{W} / \sqrt{\text{S.G.} (\Delta P)}$

$\dot{W}$  = flow-rate of test fluid, lb/sec  
S.G. = specific gravity of test fluid  
 $\Delta P$  = resultant pressure loss, psi

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Report AFRPL-TR-66-230

### III, B, Engineering Investigation (cont.)

from the tests with  $N_2O_4$  were used during the test program with  $H_2O_2$  to minimize redundant testing; however, each propellant combination was considered separately, taking into account the possibility that conditions favorable to the ignition and efficient combustion of  $N_2O_4$ /Beryllizine-21 may not be the same as those deemed satisfactory for use with the  $H_2O_2$ /Beryllizine-28 system.

(U) Ignition and combustion technology was divided into three phases: (1) engineering, (2) fabrication, and (3) test program. The work accomplished in each of these areas is discussed in the following paragraphs.

#### a. Engineering

(U) The engineering phase included the analysis of propellant performance and the generation of other data required for the design, fabrication and testing of the experimental hardware for the propellant investigations. The significant items of engineering are discussed in the following paragraphs.

#### (1) Theoretical Performance Calculations

##### (a) $N_2O_4$ /Beryllizine-21

##### 1 Evaluation of Computer Program

(C) The theoretical performance of the  $N_2O_4$ /Beryllizine system was studied previous to this program; however, a change in the heat of formation of the oxidizer and the availability of new thermodynamic data for several beryllium species necessitated a re-evaluation of the system. Preliminary to the computation of new data, an extensive examination was made of the minor species that may occur in the combustion products. The new species considered were  $BeO(g)$ ,  $(BeO)_2(g)$ ,  $(BeO)_3(g)$ ,  $(BeO)_4(g)$ ,  $(BeO)_5(g)$ ,  $(BeO)_6(g)$ ,  $Be_2O(g)$ ,  $BeOH(g)$ , and  $Be(OH)_2(g)$ . Performance calculations at various mixture ratios at 1000/14.7 psia were made for the  $N_2O_4$ /Beryllizine-21 system to ascertain which species are thermodynamically favored and should be included in the updated program. This study showed that  $(BeO)_5(g)$  and  $(BeO)_6(g)$  are always present in concentrations less than  $10^{-4}$  mole fraction. Consequently, they were eliminated from further consideration. All the other new species were included in the computer program as a result of the re-evaluation.

(C)  $BeOH$  was found to be the most significant new species occurring as a combustion product. The maximum concentration was found to be approximately 3.5 mol%. The effect of this species is to lower the performance of the system. Even the increase in the heat of formation of  $N_2O_4$  (liquid) from -6.8 Kcal/mole to -4.676 Kcal/mole was insufficient to prevent an overall decrease of about 0.7 sec of specific impulse at optimum mixture ratio. A decrease of approximately 190°F in the combustion temperature accompanied the decrease in specific impulse.

# CONFIDENTIAL

Report AFRPI-TR-66-230

## III, B, Engineering Investigation (cont.)

### 2 Fuel Composition

(C) The optimum beryllium loadings in hydrazine were calculated for optimum sea-level expansion at pressures of 500, 1000, and 1500 psia, using the revised computer program. The results are summarized in Table XXXIII. The optimum beryllium loadings at 500, 1000, and 1500 psia were found by graphical methods to be  $19.2 \pm 0.2$ ,  $20.9 \pm 0.2$ , and  $22.0 \pm 0.2\%$ , respectively. The changes in thermodynamic data and the addition of new species did not result in a significant shift in the optimum beryllium loadings or optimum mixture ratios that were calculated previously. Beryllizine containing 21% of beryllium powder was selected for use in the program.

### 3 Calculation of Detailed Performance Data

(C) Theoretical performance data were calculated for the  $N_2O_4$ /Beryllizine-21 propellant combination at chamber pressures of 500, 1000 and 1500 psia and mixture ratios of 0.40, 0.47, 0.536, 0.75, 1.00, 1.25 and 1.50. These served as basic design data and were the basis for comparison in the evaluation of experimental data. These performance data are summarized in Table XXXIV and presented graphically in Figure 24.

### (b) $H_2O_2$ /Beryllizine-28

#### 1 Fuel Composition

(C) The theoretical performance of the 98%  $H_2O_2$ /Beryllizine system had been studied prior to this program to obtain optimum concentrations of the metal in the fuel and theoretical performance values over a range of chamber pressures. Even through these data should have been recalculated, taking the new species of combustion products into account, the revised data were not considered essential for engineering design purposes. The unrevised data calculated at 1000 psia were assumed to be valid for injector design purposes since the system is similar to  $N_2O_4$ /Beryllizine and the changes in the computer program did not significantly affect the optimum beryllium loading or optimum mixture ratio for the  $N_2O_4$ /Beryllizine-21 system. Table XXXV presents data on the optimization of fuel composition for the 98%  $H_2O_2$ /Beryllizine system at 1000 psia.

#### 2 Calculation of Detailed Performance Data

(U) To provide a theoretical basis for evaluating experimental performance data from the 98%  $H_2O_2$ /Beryllizine-28 firings, the theoretical performance parameters of the propellant combination were computed. The computations covered chamber pressures of 500, 1000 and 1500 psia, mixture ratios of 0.40, 0.47, 0.529, 0.75, 1.00 and 1.25, and area ratios to approximately 1000. From these data the theoretical performance at



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Report AFRPL-TR-66-230

TABLE XXXIII

SPECIFIC IMPULSE AND COMBUSTION TEMPERATURE  
AS FUNCTIONS OF BERYLLIUM CONTENT  
IN THE  $N_2O_4/N_2H_4 + Be$  SYSTEM (C)

MR EQUIVALENT TO  $BeO, N_2, H_2$  STOICHIOMETRY

$$P_e = P_g = 14.7 \text{ psia}$$

	<u>% Be in <math>N_2H_4</math></u>	<u><math>I_s</math>, sec</u>	<u><math>T_c</math>, °R</u>
$P_c = 500 \text{ psia}$	16.67	302.22	5459
	17.00	302.51	5498
	19.00	303.41	5713
	21.00	302.97	5898
	23.00	301.61	6065
$P_c = 1000 \text{ psia}$	17.00	323.99	5568
	19.00	326.08	5803
	21.00	326.62	6007
	23.00	326.06	6190
	25.00	324.7	6354
$P_c = 1500 \text{ psia}$	19.00	337.51	5850
	21.00	338.76	6067
	23.00	338.76	6260
	25.00	337.78	6433
	27.00	336.09	6594

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XXXIV

SUMMARY OF PERFORMANCE CALCULATIONS,  
N<sub>2</sub>O<sub>4</sub>/BERYLLIZINE-21 (u)

<u>MR</u>	<u>T<sub>c</sub>, °R</u>	<u>I<sub>s</sub>, sec</u>	<u>ρ<sub>B</sub>(1)</u>	<u>I<sub>s</sub>, p</u>	<u>c*, fps</u>	<u>C<sub>F</sub></u>
<b>P<sub>c</sub> = 500 psia</b>						
0.40	5081	291.22	1.187	345.68	6196	1.512
0.47	5465	297.18	1.196	355.43	6139	1.557
0.536	5897	302.96	1.205	365.07	6277	1.553
0.75	6230	296.48	1.229	364.37	6181	1.543
1.00	6466	289.61	1.251	362.30	6059	1.538
1.25	6600	283.49	1.269	359.75	5938	1.536
1.50	6631	277.68	1.284	356.54	5810	1.537
<b>P<sub>c</sub> = 1000 psia</b>						
0.40	5128	312.23	1.187	370.62	6342	1.584
0.47	5528	319.88	1.196	382.58	6151	1.673
0.536	6007	326.63	1.205	393.59	6307	1.666
0.75	6367	320.57	1.229	393.98	6216	1.659
1.00	6627	313.99	1.251	392.80	6097	1.657
1.25	6780	307.49	1.269	390.20	5977	1.655
1.50	6822	301.19	1.284	386.73	5855	1.655
<b>P<sub>c</sub> = 1500 psia</b>						
0.40	5195	322.88	1.187	383.26	6334	1.458
0.47	5560	331.34	1.196	396.28	6157	1.504
0.536	6067	338.75	1.205	408.19	6320	1.490
0.75	6439	332.94	1.229	409.18	6231	1.477
1.00	6719	326.45	1.251	408.39	6119	1.496
1.25	6886	320.47	1.269	403.86	6003	1.485
1.50	6935	313.85	1.284	402.98	5878	1.486

(1) Bulk densities calculated at 77°F assuming ideal behavior and the following component densities: N<sub>2</sub>O<sub>4</sub>, 1.433; N<sub>2</sub>H<sub>4</sub>, 1.0036; and Be, 1.85 gm/cm<sup>3</sup>.

# CONFIDENTIAL

Report AFRPL-TR-66-230

## TABLE XXXV

SPECIFIC IMPULSE AND COMBUSTION TEMPERATURE  
AS FUNCTIONS OF BERYLLIUM CONTENT IN THE  
98%  $H_2O_2/N_2H_4$  + Be SYSTEM (C)

MR EQUIVALENT TO  $BeO$ ,  $N_2$ ,  $H_2$  STOICHIOMETRY

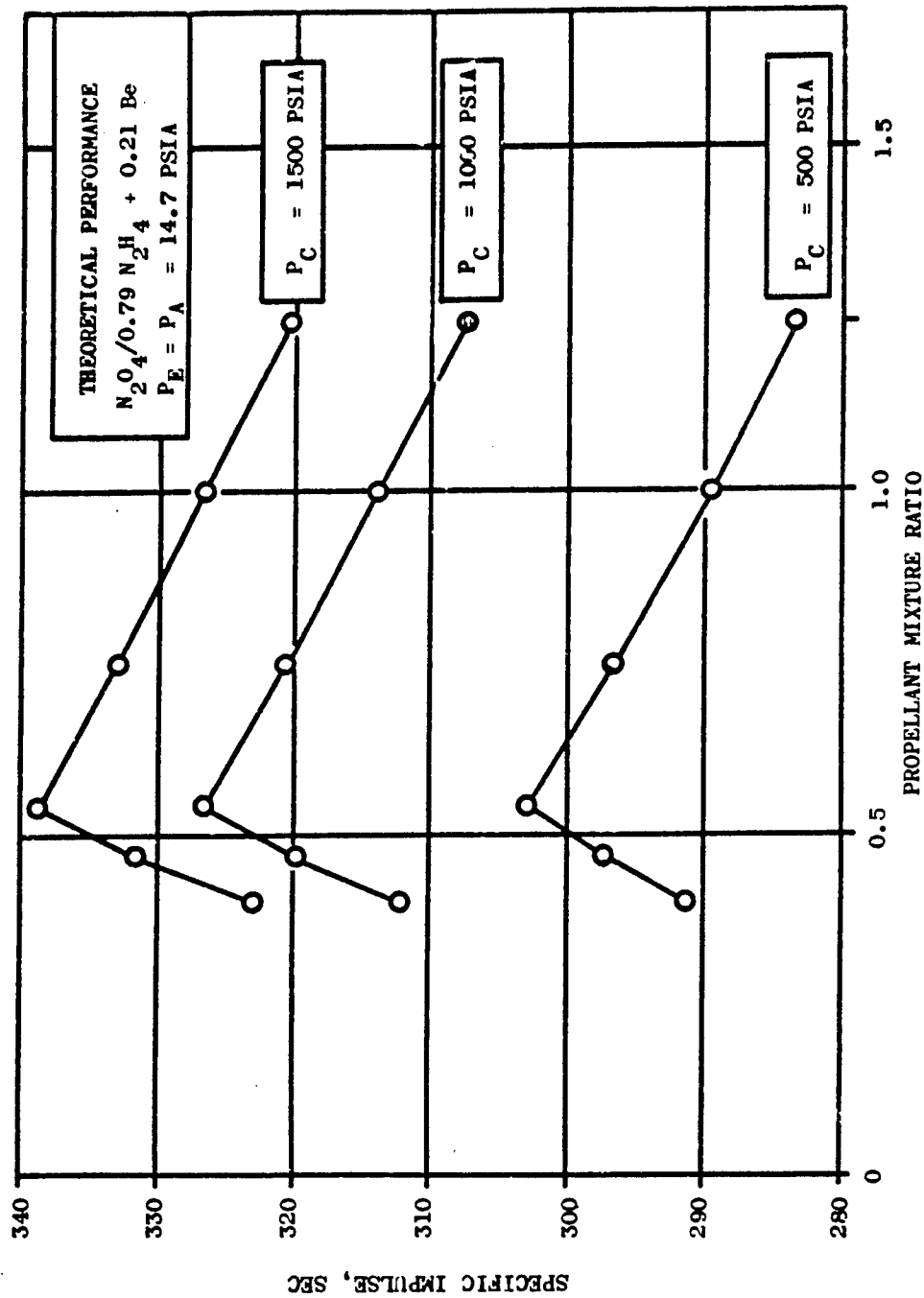
$$P_e = P_a = 14.7 \text{ psia}$$

$$P_c = 1000 \text{ psia}$$

<u>% Be in <math>H_2H_4</math></u>	<u><math>I_s</math>, sec</u>	<u><math>T_c</math>, °R</u>
10.0	312.82	5983
20.0	314.88	5843
24.5	330.20	5845
25.0	334.28	5897
28.0	335.50	6098
30.0	335.32	6217
33.0	334.10	6399
35.0	332.98	6503

CONFIDENTIAL

Report AFRPL-TR-66-230



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Figure 24. Theoretical Performance of  $\text{N}_2\text{O}_4/0.79 \text{N}_2\text{H}_4 + 0.21 \text{Be}$  (c)

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

any set of experimental firing conditions could be defined by computer interpolation, and a direct comparison of theoretical and experimental performance was readily made. The theoretical data for optimum sea level expansion and vacuum expansion through a 40:1 area ratio are summarized in Table XXXVI and plotted in Figure 25.

#### (c) Analysis of Exhaust Compositions

(C) Recently the exhaust products of the  $\text{N}_2\text{O}_4/\text{RP-1} + \text{LMH-2}$  propellant system have been analyzed and the presence of  $\text{Be}_3\text{N}_2$  has been detected and reported (Reference 7). This disclosure prompted a brief investigation to determine if the presence of this species is thermodynamically predictable in that system or in the  $\text{N}_2\text{O}_4$  or 98%  $\text{H}_2\text{O}_2$ /Beryllizine systems which are of interest in this program. To answer this question, the  $\text{Be}_3\text{N}_2$  species was incorporated into the theoretical propellant performance computer program, and equilibrium chemical compositions were computed. Calculations for the  $\text{N}_2\text{O}_4/(0.7 \text{ RP-1} + 0.3 \text{ LMH-2})$  propellant system were performed assuming a chamber pressure of 300 psia and at mixture ratios of 1.5 to 4.0 (approximate reported test conditions). These computations clearly showed that  $\text{Be}_3\text{N}_2$  should not be present in either the chamber or exhaust when the mixture ratio is greater than approximately 1.75, but should be present at somewhat lower mixture ratios. This mixture ratio is very close to  $\text{BeO}$ ,  $\text{CO}$ ,  $\text{H}_2$ ,  $\text{N}_2$  stoichiometry (1.774 is the true value), and corresponds to the demarcation between fuel-rich and oxidizer-rich regions. The experimental work (Reference 7) has shown the presence of  $\text{Be}_3\text{N}_2$  in the oxidizer-rich region (mixture ratios 1.96, 3.92, 4.53), contrary to predictions based on thermodynamic equilibrium, and indicates that the  $\text{N}_2\text{O}_4/(\text{RP-1} + \text{LMH-2})$  propellant combination under the conditions in which it was fired deviates substantially from the normal equilibrium combustion model.

(C) Similar calculations on the  $\text{N}_2\text{O}_4/\text{Beryllizine-21}$  combination at a chamber pressure of 1000 psia and mixture ratios of 0.40, 0.47, 0.536 (stoichiometric to  $\text{BeO}$ ,  $\text{H}_2$  and  $\text{N}_2$ ), and 0.75 similarly shows that  $\text{Be}_3\text{N}_2$  should be present only in the fuel-rich region (mixture ratios less than 0.536). A preliminary qualitative test of the solid exhaust products from a firing at a nominal chamber pressure of 900 psia and mixture ratio of 0.6 failed to indicate the presence of  $\text{Be}_3\text{N}_2$ . On the basis of the preliminary analyses, it does not appear that the  $\text{N}_2\text{O}_4/\text{Beryllizine-21}$  system behaves like the  $\text{N}_2\text{O}_4/(\text{RP-1} + \text{LMH-2})$  combination, but, rather, behaves more nearly as predicted by equilibrium thermodynamics. None of the differences between the combinations provides a satisfactory explanation of the apparent difference in combustion behavior, but it is likely related to a combination of hardware effects (injector configuration and chamber and nozzle size and geometry), operating conditions (pressure,  $L^*$ , etc.), and to propellant composition (carrier and form of beryllium).

# CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XXXVI

## SUMMARY OF PERFORMANCE CALCULATIONS

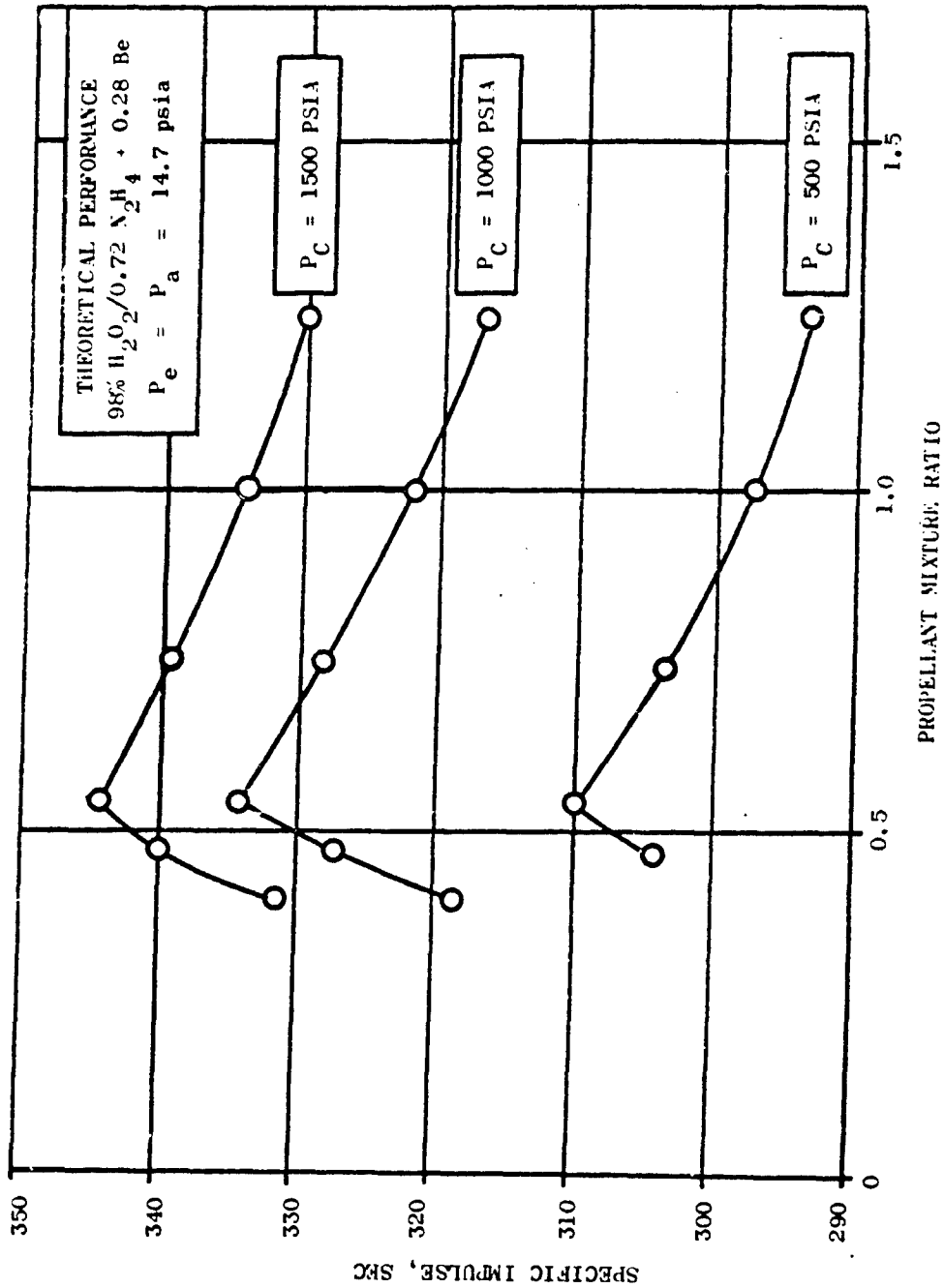
98%  $H_2O_2$ /BERYLLIZINE-28 (C)

MR	T <sub>c</sub> , °R	ρ <sub>B</sub> (1)	C*	I <sub>s</sub> Vacuum at		C <sub>F</sub> Vacuum at	
				Opt. S.L.	40:1	Opt. S.L.	40:1
P <sub>c</sub> = 500 psia							
0.40	5081	1.219	----	296.7	-----	-----	-----
0.47	5369	1.228	6236	303.8	390.7	1.567	2.015
0.529	5785	1.235	6368	309.6	400.6	1.564	2.024
0.75	5934	1.256	6271	303.5	393.2	1.557	2.017
1.00	6015	1.276	6168	297.8	386.3	1.553	2.015
1.25	6072	1.291	6082	293.3	380.7	1.552	2.014
P <sub>c</sub> = 1000 psia							
0.40	5110	1.219	----	319.1	-----	-----	-----
0.47	5423	1.228	6221	327.3	391.4	1.692	2.024
0.529	5879	1.235	6392	334.3	401.2	1.683	2.019
0.75	6034	1.256	6293	327.8	393.8	1.676	2.013
1.00	6124	1.276	6193	321.8	386.9	1.672	2.010
1.25	6189	1.291	6110	317.1	381.4	1.669	2.008
P <sub>c</sub> = 1500 psia							
0.40	5174	1.219	----	330.5	-----	-----	-----
0.47	5447	1.228	6237	339.7	391.7	1.752	2.020
0.529	5929	1.235	6404	347.0	401.4	1.743	2.016
0.75	6089	1.256	6307	340.4	394.1	1.736	2.010
1.00	6185	1.276	6208	334.3	387.3	1.732	2.007
1.25	6256	1.291	6126	329.4	381.9	1.730	2.005

- (1) Bulk densities calculated at 77°F assuming ideal behavior and the following component densities: 98%  $H_2O_2$ , 1.4309,  $N_2H_4$ , 1.0036; and Be, 1.85 gm/cm<sup>3</sup>.

CONFIDENTIAL

Report AFRPL-TR-66-230



CONFIDENTIAL

Figure 25. Theoretical Performance of 98%  $H_2O_2/0.72 N_2H_4 + 0.28 Be$  (C)

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Report AFRPL-TR-6G-230

### III, B, Engineering Investigations (cont.)

(U) Further computations on 98%  $H_2O_2$ /Beryllizine-28 have not been made to determine if  $Be_3N_2$  should appear, but from the data on  $N_2O_4$ /RP-1 + LMH 2 and  $H_2O_4$ /Beryllizine-21, it can be safely assumed that the formation of  $Be_3N_2$  is similarly thermodynamically favored only in the fuel-rich region (mixture ratios less than 0.529). It should be noted that the inclusion of the  $Be_3N_2$  species changes the theoretical performance parameters in the fuel-rich region. For example, the chamber temperature of  $N_2O_4$ /Beryllizine-21 increases approximately 8.3 and 3.7% at mixture ratios of 0.40 and 0.47, respectively, over the values previously reported (Reference 8), and specific impulses increase approximately 2.6 and 1.2% at mixture ratios of 0.40 and 0.47, respectively. At higher mixture ratios the values remain valid as previously reported. The performance parameters of 98%  $H_2O_2$ /Beryllizine-28 as given in Table XXXVI can also be anticipated to change by a similar amount at the same mixture ratios.

#### (2) Design

##### (a) Design Data

(C) The design values used in this work were based on the maximum theoretical specific impulse of the  $N_2O_4$ /Beryllizine-21 system at a chamber pressure of 1000 psia. The mixture ratio corresponding to maximum specific impulse is 0.536. For design purposes, this value was increased to 0.6 because Aerojet's experience during the Improved Titan (Titan IIA) Predevelopment Program (Reference 9) indicates that the specific impulse delivered at a slightly higher mixture ratio results in higher performance than that delivered at the optimum theoretical value.

(C) The theoretical data were corrected for design purposes by calculating an efficiency balance. The efficiencies assumed for specific impulse and thrust coefficient were 91 and 95%, respectively, based upon Aerojet's experience with the design of TCA's for evaluation of  $N_2O_4$ /Alumizine propellant systems (References 9 and 10). A combustion efficiency of 96% was obtained from these values.

(C) The theoretical values for specific impulse, characteristic velocity and thrust coefficient selected for design purposes were 326.2 sec, 6361 ft/sec and 1.650, respectively. These were multiplied by the proper coefficients of efficiency, giving 297 sec, 6120 ft/sec and 1.570. The total propellant flow rate, 3.39 lb/sec, was obtained by dividing the design thrust by the design specific impulse. The flow rates of the individual propellants were 2.12 lb/sec of fuel and 1.27 lb/sec of oxidizer at the design mixture ratio of 0.6.



## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### (b) Design Parameters

(U) A nozzle area ratio of 12:1 was calculated from theoretical data corresponding to a pressure ratio of 1000/14.7. A throat area of 0.637 sq in. was computed from the values for nominal thrust and chamber pressure and the design value for the thrust coefficient. The throat diameter was calculated to be 0.9. Using the throat area, the nozzle area ratio and their previously defined relationship, the area and diameter of the nozzle exit were found to be 7.7 sq in. and 3.12 in., respectively.

(U) A parametric analysis was performed to obtain the necessary design parameters including contraction ratio and length of the combustion chamber, using a characteristic length of 100 in. The 100-in.  $L^*$  is based on Aerojet's testing experience with gelled-metalized propellant systems at nominal chamber pressures of 500 psia (Reference 10) and 1500 psia (Reference 9). A nominal contraction ratio of 7:1 was selected, along with a chamber diameter of 2.35 in. The corresponding chamber length, measured from the face of the injector to the nozzle entrance, was calculated to be 10 in. (However, the combustion chamber length was increased to 12.5 in. during the test program.)

(U) When the parameters for the design of the thrust chamber had been established, detailed engineering calculations were performed and the preliminary design phase of the engineering task was begun.

#### (c) Injectors

(C) A major problem associated with the use of heterogeneous propellants containing beryllium is obtaining ignition and efficient combustion of the metal particles. It is generally accepted that beryllium particles are coated with oxide shells which must melt, or become porous, before the elemental beryllium will ignite. Since the oxide melting temperature is 5080°R, successful ignition should occur only when the temperature of the oxide approaches that value. Unfortunately, the temperature apparently required for ignition of beryllium is unobtainable from the reaction of the hydrazine constituent in Beryllizine with either  $N_2O_4$  or 98%  $H_2O_2$  within the range of mixture ratios of interest. A major program objective was the development of a technique for ignition of the beryllium constituent of Beryllizine at mixture ratios near optimum.

(C) Early in the program, several solutions to the problem of igniting the beryllium in the combustion chamber near the optimum mixture ratio of the propellant systems under study were considered. The first approach studied was modification of the beryllium powder to promote ignition. Particle coating with either aluminum or chromium appeared promising,

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

as did particles manufactured from an alloy of beryllium with another metal, such as magnesium. Small-diameter beryllium particles, and particles manufactured in an inert atmosphere just prior to fuel mixing were also considered for application. Another approach studied was modification of the fuel formulation, such as physical mixtures of beryllium and aluminum powders, to solve the problem of beryllium ignition. Slug-starting with a third fluid, pyrotechnic igniters and starting at high mixture ratio were other solutions given consideration. The final approach studied was tailoring part of the injector geometry to produce conditions favorable to the ignition of the beryllium particles while operating at an overall mixture ratio near optimum.

(C) Aerojet first suggested a solution to the problem of ignition and combustion of the beryllium in 1963 (Reference 10). The concept consisted of an injector with a single injection element operating at a mixture ratio favorable for ignition of the metal particles. The balance of the injection elements would operate at such a mixture ratio as to result in an overall ratio favoring high specific impulse. It was postulated that if ignition was achieved and the igniting element allowed to remain functioning, ignition would propagate throughout the zone of combustion and would be sustaining. The degree of efficiency would then be a function of the internal geometry of the combustor.

(C) The solution of the ignition and combustion problem was a dominant consideration in the design of the two types of injectors used in the ignition and combustion investigation. That is to say, the injectors were designed to produce locally, through control of mixture ratio, the composition and temperature conditions that are believed to be most favorable for achieving ignition and efficient combustion of the beryllium particles and, at the same time, to promote a high overall efficiency at or near the theoretically optimum mixture ratio. To achieve these somewhat diverse conditions with the same injector, a hot-core injector concept was adopted. This concept and the conditions within the hot core that influenced injector design are described in the following paragraphs.

(C) To aid in defining the conditions of temperature and composition (mixture ratio) most favorable for achieving beryllium ignition and combustion, a literature review was conducted. On the basis of a number of reports covered in the review (References 11 through 16), it was concluded that the temperature and the water vapor concentration should be as high as possible. The actual temperature requirements suggested vary from 2920°R in a pure water vapor environment to 5155°R in a pure oxygen atmosphere (Reference 16) to approximately 6000°R in an atmosphere of propellant reaction products having oxidizing capabilities (Reference 17). To define the operating conditions under which  $N_2O_4$ /Beryllizine-21 will yield maximum temperature and water vapor concentration prior to beryllium ignition, it was postulated that

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

$N_2O_4$  and the  $N_2H_4$  contained in Beryllizine-21 will ignite spontaneously upon contact, burn completely, and come to thermal equilibrium with elemental beryllium. Calculations based upon this postulation show that both temperature and water vapor concentration become maximum under these circumstances at a mixture ratio ( $N_2O_4$ /Beryllizine-21) of approximately 1.15. At this mixture ratio the flame temperature is approximately 5200°R and the concentration of water in the vapor phase is approximately 50 mol%. Thus, a mixture ratio of 1.15 was selected as the most favorable for achieving initial beryllium ignition.

(C) Once beryllium combustion is established, the flame temperature theoretically increases to approximately 6730°R and the water concentration in the vapor phase decreases to approximately 25 mol%. These conditions appeared sufficient for promoting continued combustion of incoming propellants. Unfortunately, the operation of  $N_2O_4$ /Beryllizine-21 at such a high overall mixture ratio (1.15) would theoretically limit the achievable performance to approximately 95% of that possible at the optimum mixture ratio (0.536). Because such a performance degradation is unacceptable, attention was focused on selecting an overall mixture ratio that would provide maximum delivered specific impulse (considering both probable combustion efficiency and theoretical performance) and still permit operation, at least locally, under conditions most favorable for beryllium ignition and flame propagation.

(C) Extensive experience (Reference 9) has shown that maximum specific impulse is obtained with  $N_2O_4$ /Alumizine at mixture ratios slightly higher, perhaps 10%, than that theoretically predicted. Assuming the same situation can be anticipated with  $N_2O_4$ /Beryllizine-21, an overall mixture ratio of 0.6 (112% of the optimum theoretical value) was selected for engineering design purposes. To permit operation with a locally high mixture ratio (1.15) to favor beryllium ignition and propagation, and still obtain an overall mixture ratio of approximately 0.6 to favor maximum delivered specific impulse, the hot-core injector concept was adopted. In this concept, approximately 15% of the total propellant was used to establish a hot-core by operating at a mixture ratio near 1.15, while approximately 85% of the total propellants entered the chamber at a mixture ratio near 0.536 (the theoretically optimum value). The combustion products from the two zones intermixed as they passed down the combustion chamber and formed a zone of uniform composition at an overall mixture ratio of 0.6.

(C) Two types of injectors were designed for the ignition and combustion investigations, both based on the hot-core principle discussed previously. One is a modified version of the Centrojet injector developed at the U.S. Naval Ordnance Test Station (Reference 18). The other is based on a concept developed by Aerojet during the Improved Titan Predevelopment Program (Reference 9). In each of these designs, the orifices in the

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

center of the injector were sized to give a high mixture ratio to provide a hot-core that would be favorable to the ignition of the beryllium. The orifices toward the perimeter of the face were, therefore, sized to give a mixture ratio such that the overall ratio was 0.6.

(C) The pattern of the peripheral area of the Centrojet injector (see Figure 26) consisted of a single circle of 44 alternating fuel and oxidizer orifices. The orifices were drilled in showerhead fashion near the inner wall of the combustion chamber. Each orifice was fed with propellant from a feed-hole drilled from the appropriate manifold within the body of the injector. The injected streams of propellant were converged to a single point in the combustion chamber by a mixing and deflecting ring located on the injector face. The ring did not act as a splash-plate, but caused the streams of propellant to impinge as they approached the point of convergence, where they mixed.

(C) The original Centrojet injector had a small nozzle located in the center of the injector face. Part of the gas from the gas generator, which was used for pressurization of the propellant tanks, entered the chamber through this nozzle and provided a second means for propellant mixing. To provide the hot-core for the ignition of beryllium, this nozzle was replaced by a second liquid-injection area. The pattern for this area was based upon a concept that had been successfully demonstrated at the Air Force Rocket Propulsion Laboratory (Reference 19). The pattern consisted of a central swirler that produced a hollow cone of fuel. The fuel cone was impinged upon by fans of oxidizer resulting from the impingement of streams from six oxidizer doublets arranged around the swirler. After ignition, the direction of flow of burning propellants was axial and they impinged on the inner wall of the hollow cone of propellants converging from the peripheral deflector ring.

(C)) The injector face was protected from the high temperature of combustion (6150°R at the design mixture ratio) by transpiration cooling. Initially, 5% of the oxidizer was distributed over the entire area of a porous face, but was later increased because of injector face burning. Aerojet has demonstrated the concept of protecting injectors from the effects of Alumizine and  $N_2O_4$  combustion by transpiration cooling through a porous face (Reference 9). The oxidizer cooling not only protects the face from high temperature, but also prevents recirculation of the hot gases and burning particles which, if not prevented, results in extensive damage to the injector face.

(C) In the second injector designed for use in this program (Figure 27), the pattern for the hot-core area was a pentad. Four streams of oxidizer impinged at a 45° angle on a central fuel stream. All the remaining oxidizer entered the chamber through a porous element that covered

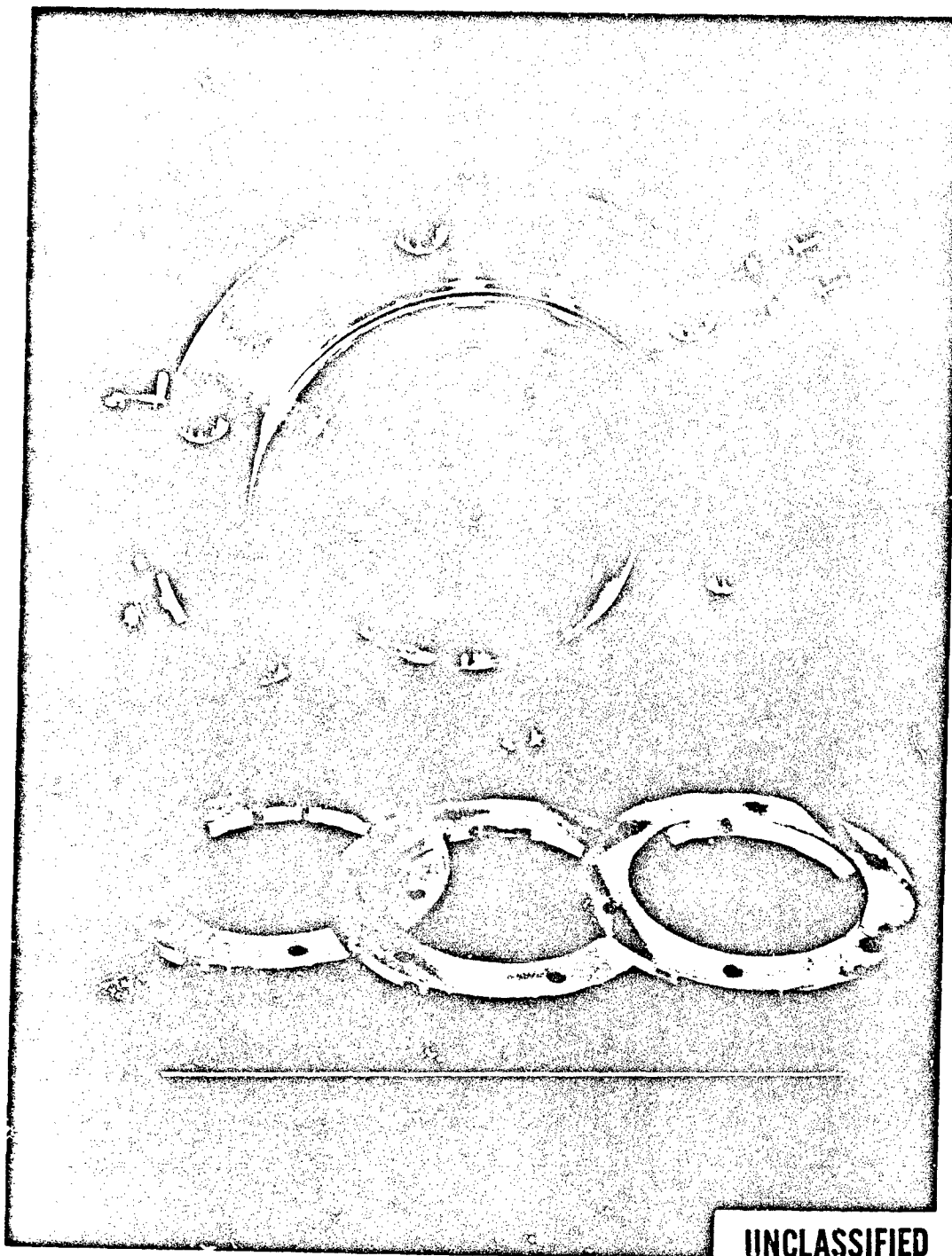


Figure 26. Modified Centrojet Injector

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Report AFRPL-TR-66-230

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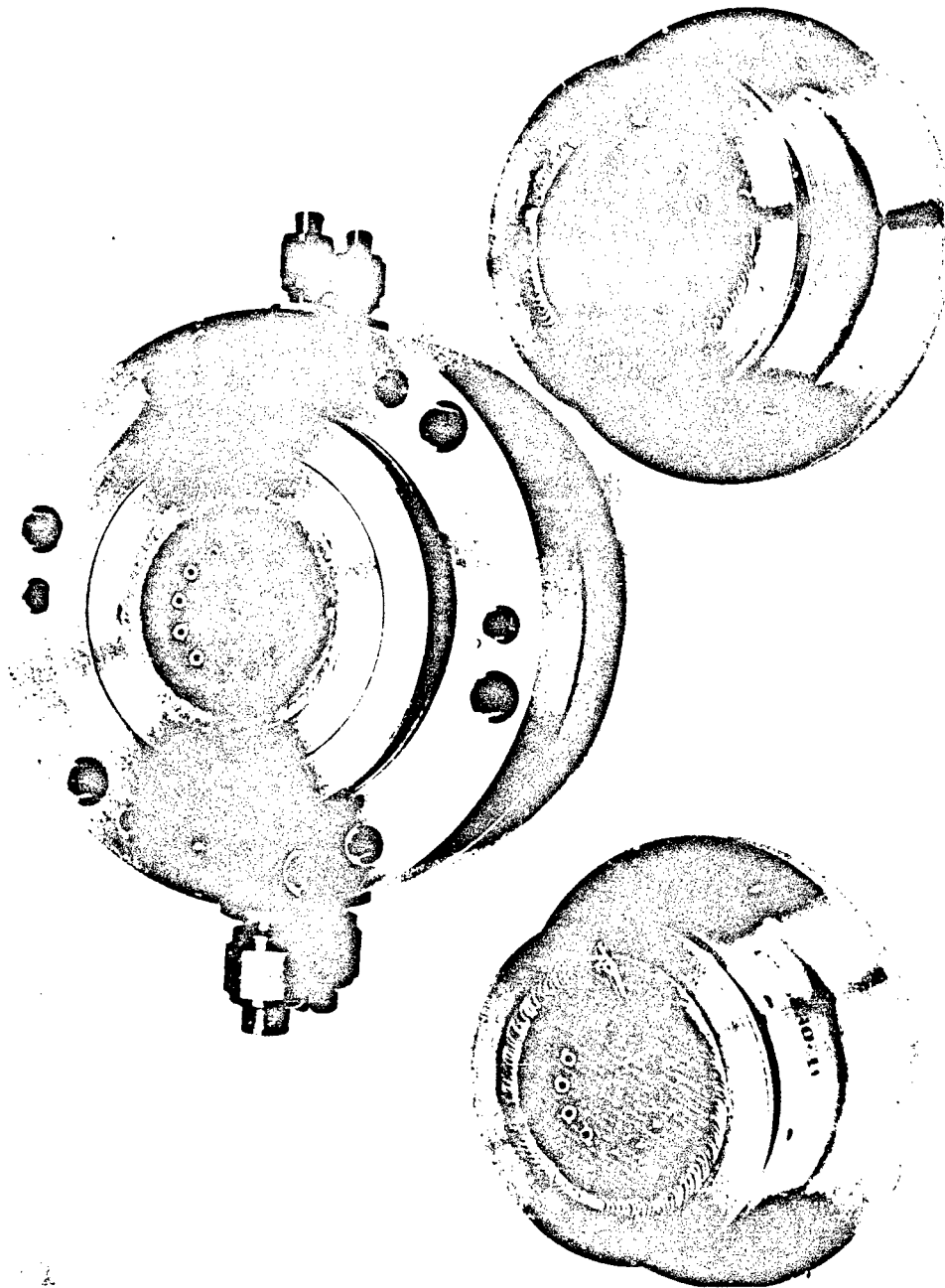


Figure 27. Injector Assembly, Second Design

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## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

covered the face of the injector. The fuel not injected into the hot-core was injected through 12 orifices that were arranged in a circle peripheral to the pentad element. In one of the three versions of this design, these 12 orifices injected fuel parallel to the axis. In the other two, the orifices injected fuel at 15 and 30° angles to the axis to effect impingement downstream from the point of impingement of the pentad element.

#### (d) Combustion Chamber

(U) The combustion chamber was constructed in segments (Figure 28). Each segment consisted of an outer Type 347 stainless-steel shell surrounding an insert of ablative material. The inserts were fabricated from Fiberite MX-2646 phenolic material reinforced with Refrasil and compression-molded to a density of 105 lb/cu ft. The inserts were coated with RTV-60 silicone rubber and placed in the steel shells, heated to 200°F. After cooling, the fit between the outer shells and the ablative inserts was 0.002 in. or less. A primary seal, in the form of a ring machined from laminated Fiberglas, was placed at the interface of all ablative inserts to prevent the hot products of combustion from damaging the inner walls of the shells. All joints in the combustion chamber were sealed with RTV-60 silicone rubber, and zinc chromate paste was used as a sealant at the interface of the injector with the chamber. Ports for the measurement of chamber pressure were located in each segment of the combustion chamber to permit measurements of pressure at various points along the chamber. The segments of the combustion chamber were easily refurbished by removing the old insert and installing a new insert as described above.

#### (e) Nozzle

(U) The composite nozzle (Figure 28) consisted of five components, the throat, a convergent section, a divergent section, an O-ring for a throat seal, and a seal ring.

(U) The convergent section of the nozzle was fabricated from Fiberite MX-2646 ablative material, compression-molded to a density of 105 lb/cu ft. Previous testing with Alumizine and  $N_2O_4$  (Reference 9) has shown that a nozzle entrance fabricated from Fiberite MX-4926 phenolic material, reinforced with carbon cloth, is durable for test durations up to 10 sec. Nevertheless, Fiberite MX-2646, which is less expensive than the material reinforced with carbon cloth, was chosen because the duration of the tests was only 2 sec and the inserts could be replaced between tests, if necessary.

(U) Inserts for the divergent section of the nozzle were also fabricated from Fiberite MX-2646 for the same reasons discussed in the previous paragraph. These were used with ATJ graphite throat inserts during the tests.

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Report AFRPL-TR-66-230

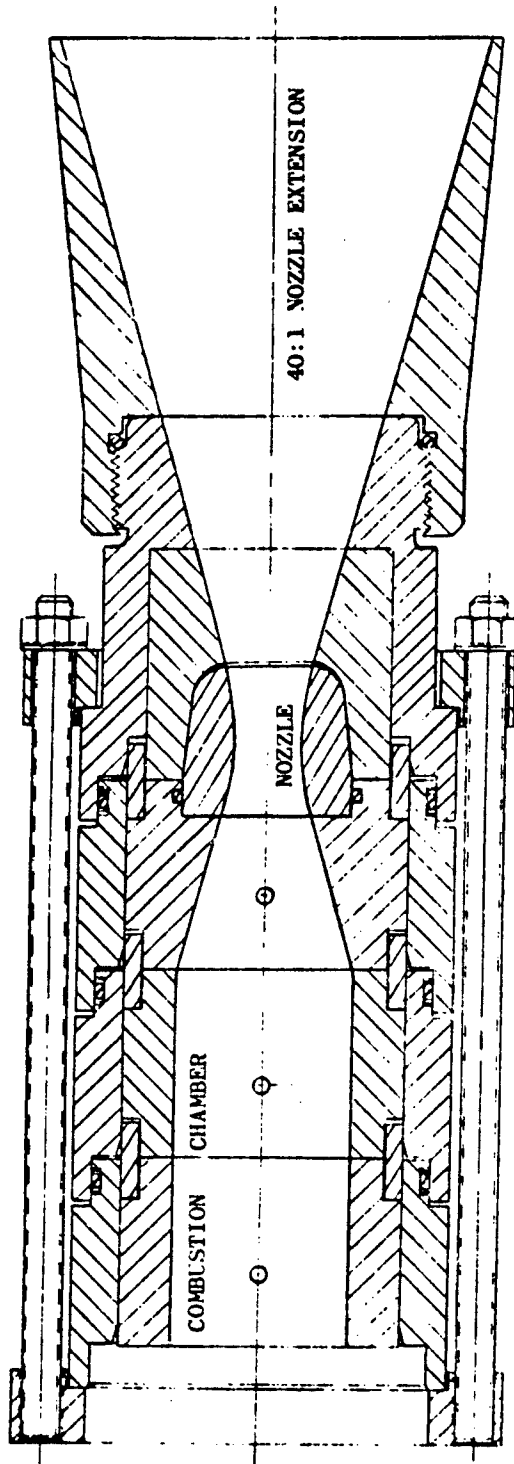


Figure 28. Uncooled Thrust Chamber

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) An O-ring was placed around the throat insert to prevent gas from leaking past the throat. The seal material was butyl rubber and provided a gas seal. As an additional precaution, silicone rubber was used at each end of the throat insert for primary seals. The seal ring afforded additional protection for the nozzle shells from the hot products of combustion.

(U) The nozzle components were inserted into the two nozzle shells (Figure 28). The shells were fabricated from Type 347 stainless steel. The nozzle extension was coated with zirconium oxide over a 0.005-in. nichrome base. Both the coating and the base were applied with a commercial plasma torch. The method of assembly was the same as that for the combustion chamber. The shells were heated to approximately 200°F and the nozzle components, coated with RTV-60 and catalyst, were dropped into place. After cooling, the throat O-ring and seal ring were installed and coated with the silicone rubber. After the outer O-ring and retainers were in place, the throat was installed and the shells were pressed together to complete the assembly. The nozzle was now ready to be mated with the combustion chamber.

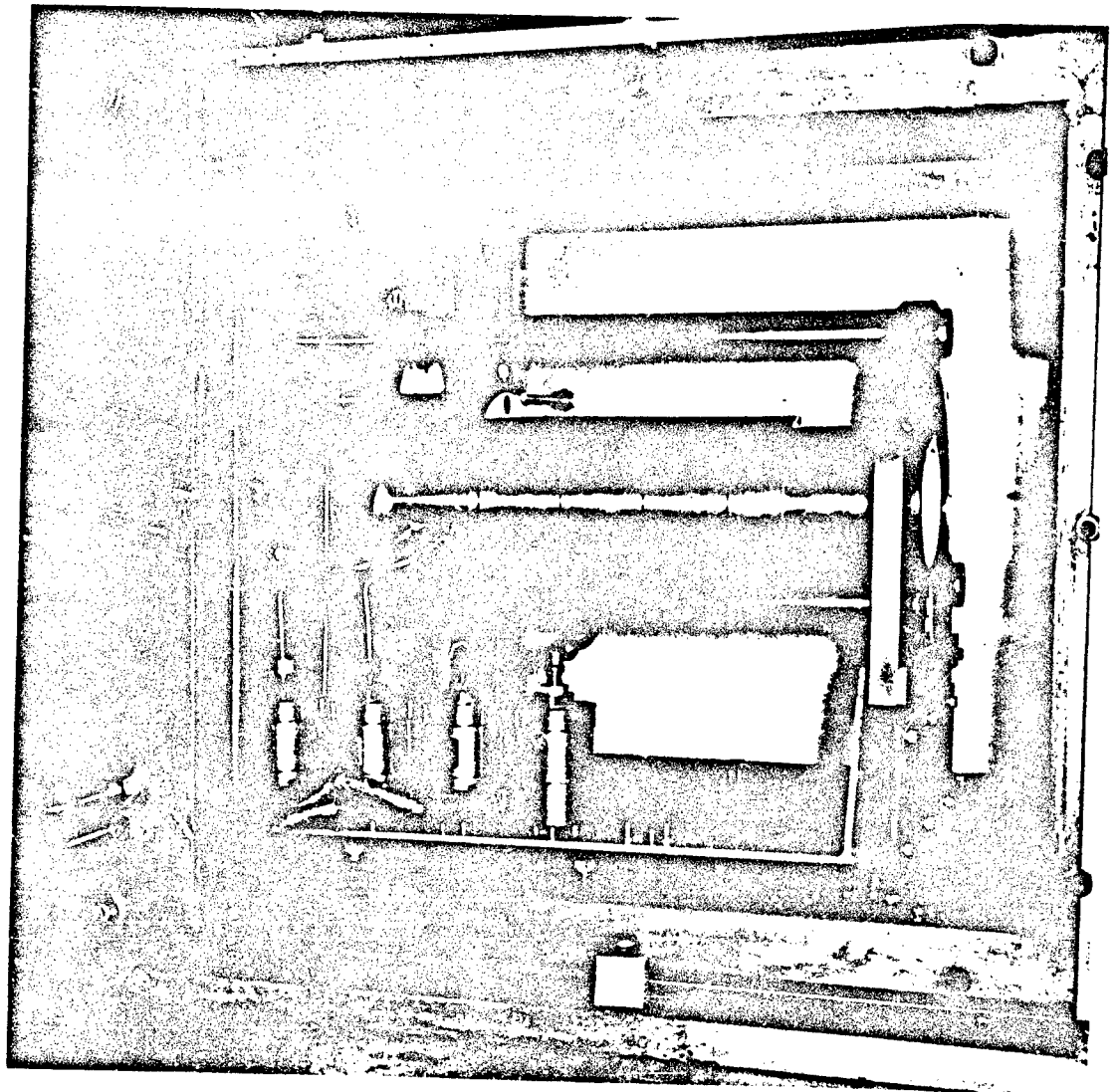
#### (f) Thrust Chamber Assembly

(U) The thrust chamber assembly consisted of the injector, combustion chamber, nozzle assembly, and stringer bolts assembled as shown in Figure 29. The O-ring groove located on the outside near the end of the nozzle was for a cover, which enabled leak-checking of TCA joints and the propellant feed systems prior to testing.

(U) During the test program the thrust chamber assembly was modified by increasing the nozzle area ratio to 40:1. Originally, the area ratio of the nozzle was selected for local ambient conditions; however, since all tests were conducted in conjunction with a vacuum catch tank, the area ratio was increased to demonstrate performance under altitude conditions. Figure 30 depicts the thrust chamber assembly mounted on the test stand with the nozzle extension in place.

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Report AFRPL-TR-66-230



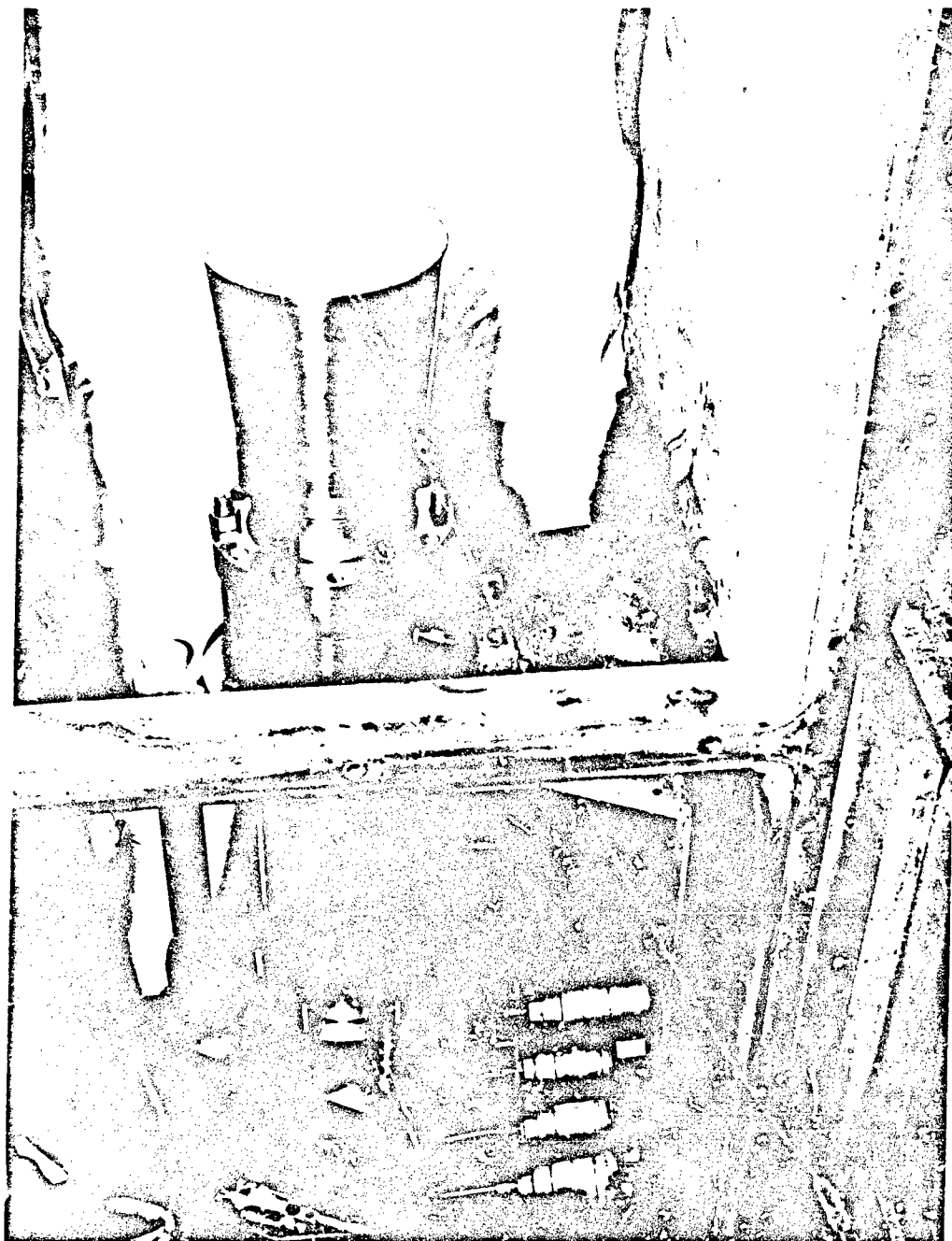
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Figure 29. Test Stand and Thrust Chamber Assembly

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Report AFRPL-TR-66-230



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Figure 30. Test Stand and Thrust Chamber Assembly with 40:1 Area Ratio Nozzle

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### b. Fabrication

##### (1) Injectors

(U) Initially, one injector of each of the two designs was procured. Enough spare parts for each were included to ensure that the initial tests could be accomplished even in the event of a major failure. After the preferred design was selected, based upon the results of testing, enough injector components were procured to complete the program.

(U) The modified Centrojet Injector, along with the three rings for deflecting and mixing the streams of propellant, is shown in Figure 26. The second injector, along with the two inserts for pattern variation, is shown in Figure 27. After receipt from the vendors, the units underwent hydraulic testing with water as part of the inspection procedure. This testing provided verification of structural integrity, water calibration of the various flow circuits and pattern checks. Tables XXXI and XXXII contain the hydraulic data for the injectors obtained from the hydraulics laboratory and compare the design values with the results obtained from hydro-testing and the initial firings. The tables also present comparisons of the hydraulic factors ( $K_v$ ) for the injector fuel circuits.

##### (2) Thrust Chambers

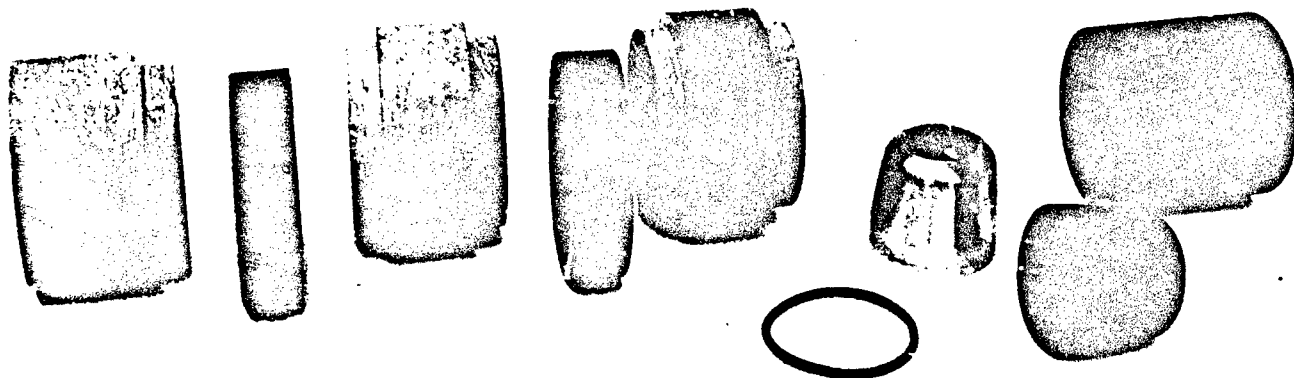
(U) Three complete thrust chamber assemblies were procured, along with additional sets of inserts. When the durability of the materials of construction had been verified, enough inserts were procured to complete the program.

(U) Figure 31 shows the inserts, unassembled, for a short thrust chamber. The inserts shown are the Refrasil-reinforced-phenolic inserts for the combustion chamber and nozzle. Between these inserts are shown the Fiberglas seal rings. Two throats are shown, one of ATJ graphite and the other of tungsten infiltrated with silver. The ATJ graphite throats were planned for all tests and several silver-tungsten inserts were furnished to the program for evaluation when possible (these inserts were not tested). The O-ring is a throat seal which prevents gas from leaking past the throat. A nozzle divergence of ATJ graphite is shown but the majority of these inserts were fabricated from the phenolic material reinforced with Refrasil. Figure 32 shows these components assembled.

(U) The metallic components are shown in Figure 30. The components are assembled into a thrust chamber and along with the modified Centrojet injector are shown on the test stand.

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Report AFRPL-TR 66-230



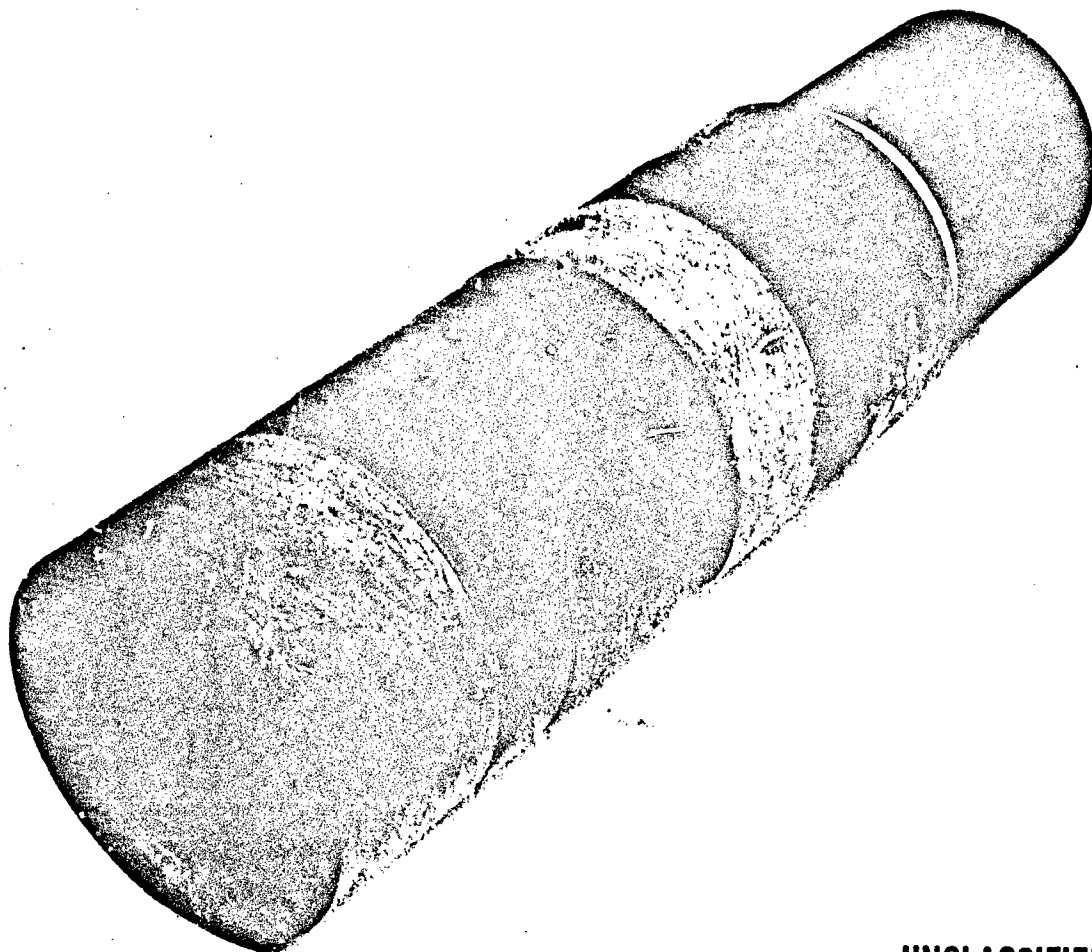
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Figure 31. Inserts, Thrust Chamber

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Report AFRPT-TR-65-230



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Figure 32. Inserts Assembled, Thrust Chamber

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Report AFRIL-TR-66-230

### III, B, Engineering Investigations

#### c. Test Program

##### (1) Test Facility

(U) The experimental portion of the program was performed using existing facilities of the Physics Laboratory, Research and Technology Operations. A test system, comprised of several subsystems, was constructed around an existing concrete-thrust block located in a test cell. A junction box for control and instrumentation connections and piping for nitrogen gas were previously installed in the cell. A description of each subsystem is presented in the following paragraphs.

##### (a) Thrust Mount

(U) The thrust mount, Figure 33, was basically a carriage mounted on two flexure supports of 1/16 in. stainless steel. The load cell (not shown) was mounted on the axis of the thrust chamber behind the injector. The load cell was affixed to a stationary support attached to the concrete-thrust block. A standard load cell and a hydraulic cylinder were mounted in series with the load cell used to gather thrust data during testing. The design of the mount was such that neither the standard cell nor the hydraulic cylinder was in compression or tension during testing.

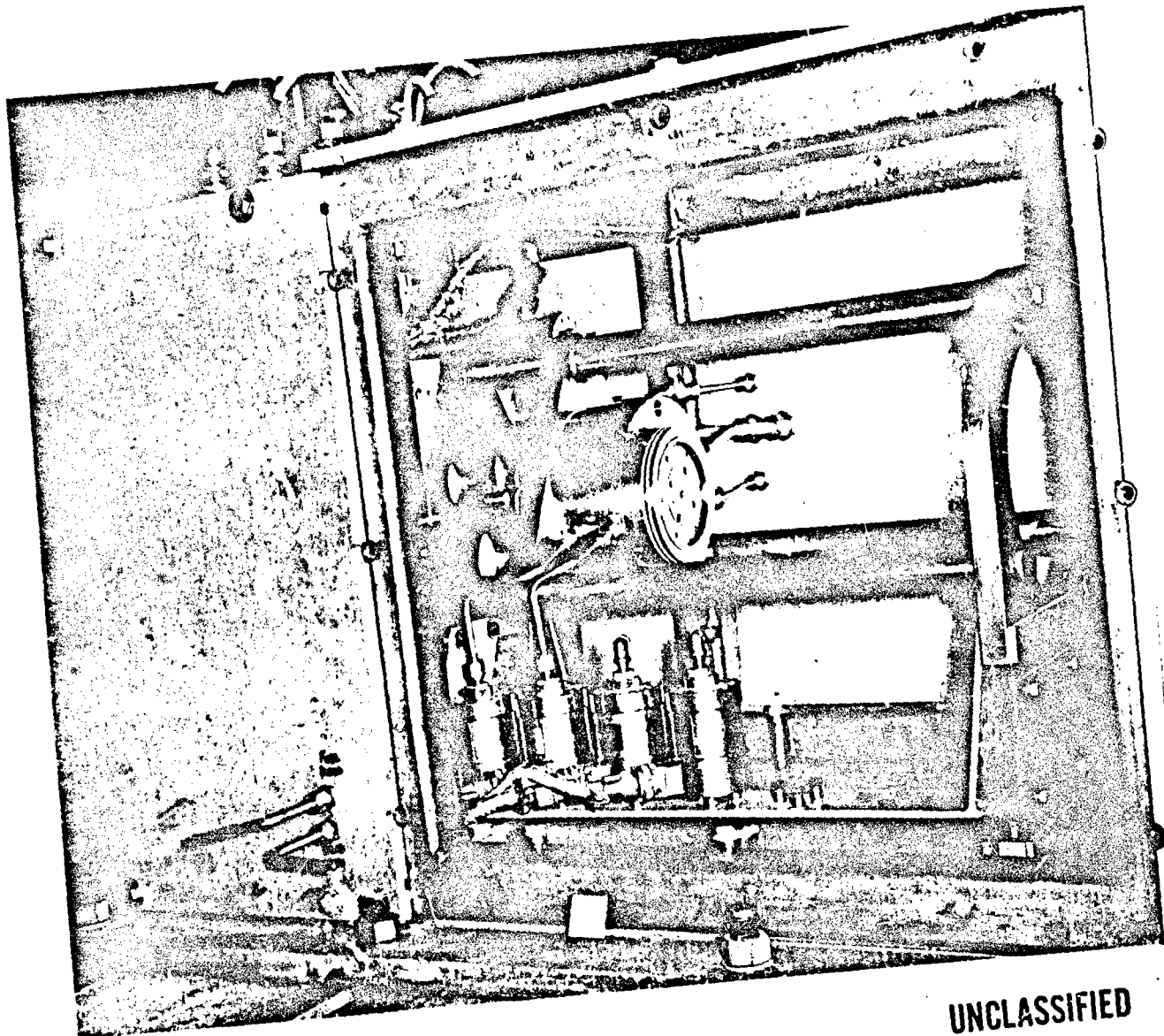
##### (b) Vacuum System

(U) A vacuum catch tank, Figure 34, was used to trap the toxic exhaust products. The figure shows the tank, which is approximately 100 ft long and 12 ft in diameter, the 3-ft gate valve and the end of the 3-ft-diameter exhaust duct. Also shown is the water deluge line leading into the tank. Figure 35 is a view of the test stand and of the exhaust duct. Figure 36 shows the bellows and the thrust stand enclosure around the thrust chamber assembly. The enclosure remained sealed from the atmosphere except when test hardware was being exchanged. This procedure relieved the problems caused by beryllium contamination. The use of a bellows eliminated any problem caused by misalignment of the exhaust duct with the test stand enclosure at the time of installation or by subsequent thermal expansion and contraction. The entire system is designed to be evacuated to a pressure of less than 1 psia. The gate valve was included so that the thrust chamber assembly could be replaced while the tank was being evacuated.

##### (c) Oxidizer Feed System

(C) The oxidizer feed system, Figure 37, was designed and fabricated for use with either  $N_2O_4$  or high-strength  $H_2C_2$ .

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Report AFFPL-TR-65-230



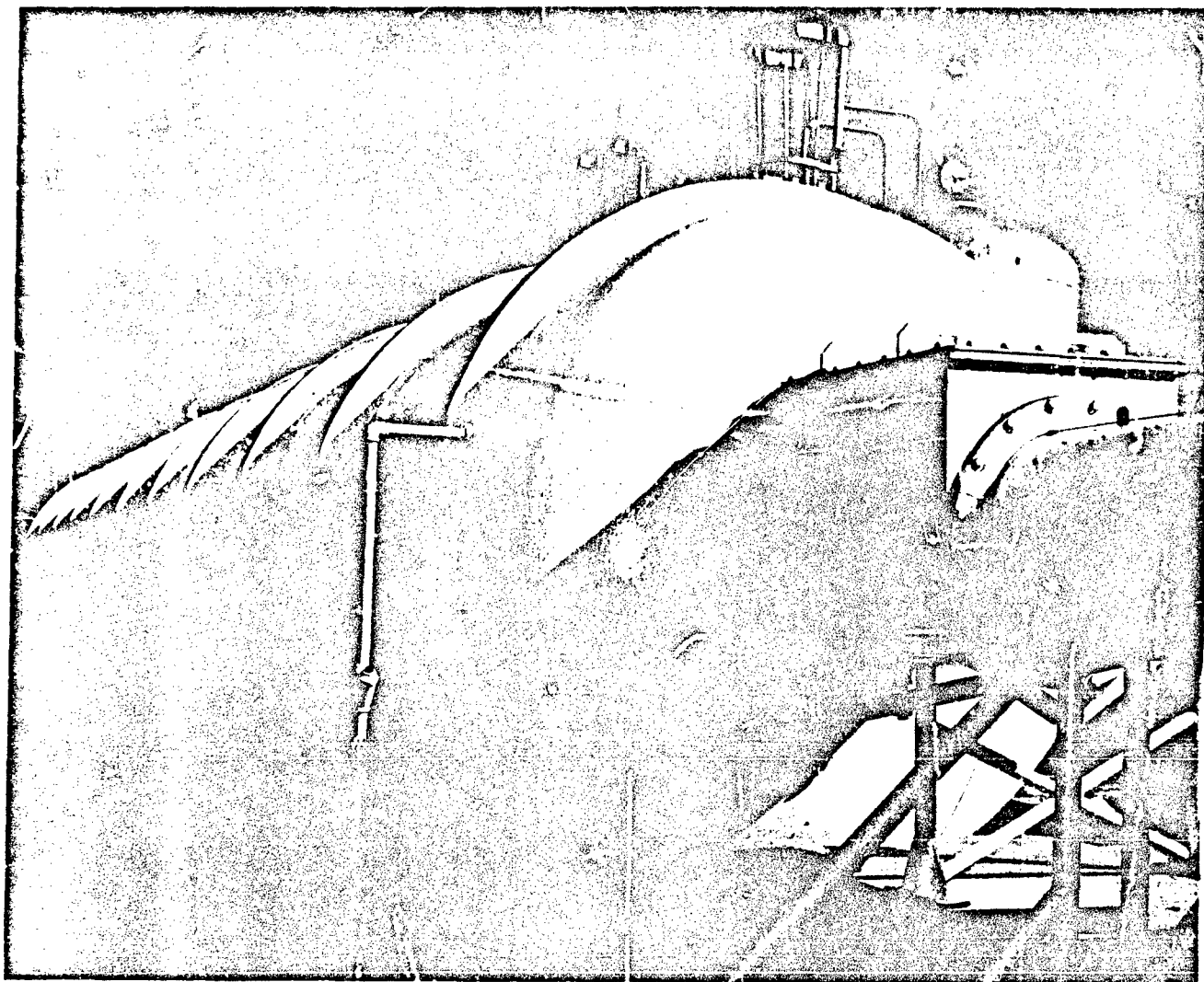
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Figure 33. Tarust Mount

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Report AFRPL-TR-66-230



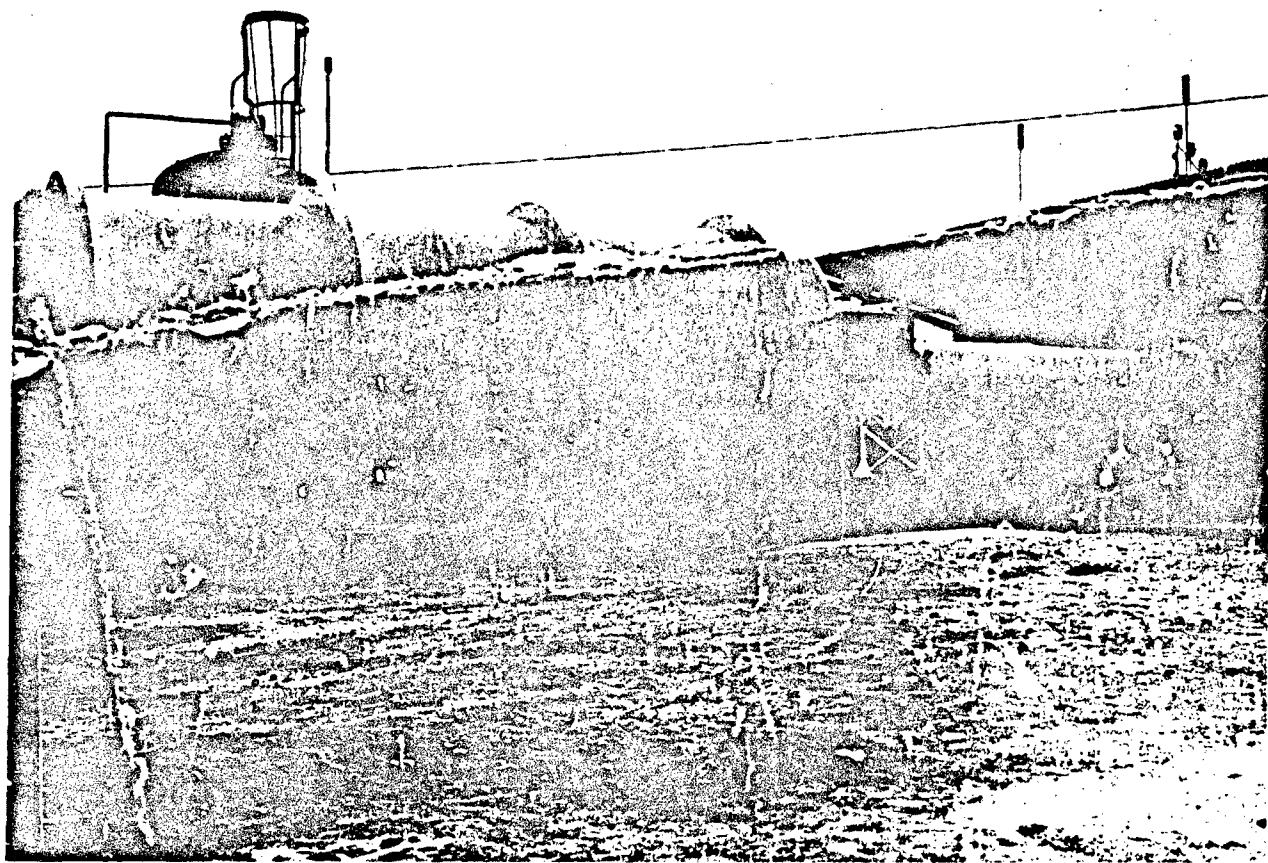
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Figure 34. Vacuum Catch Tank and Gate Valve

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Report AFRPL-TR-66-230



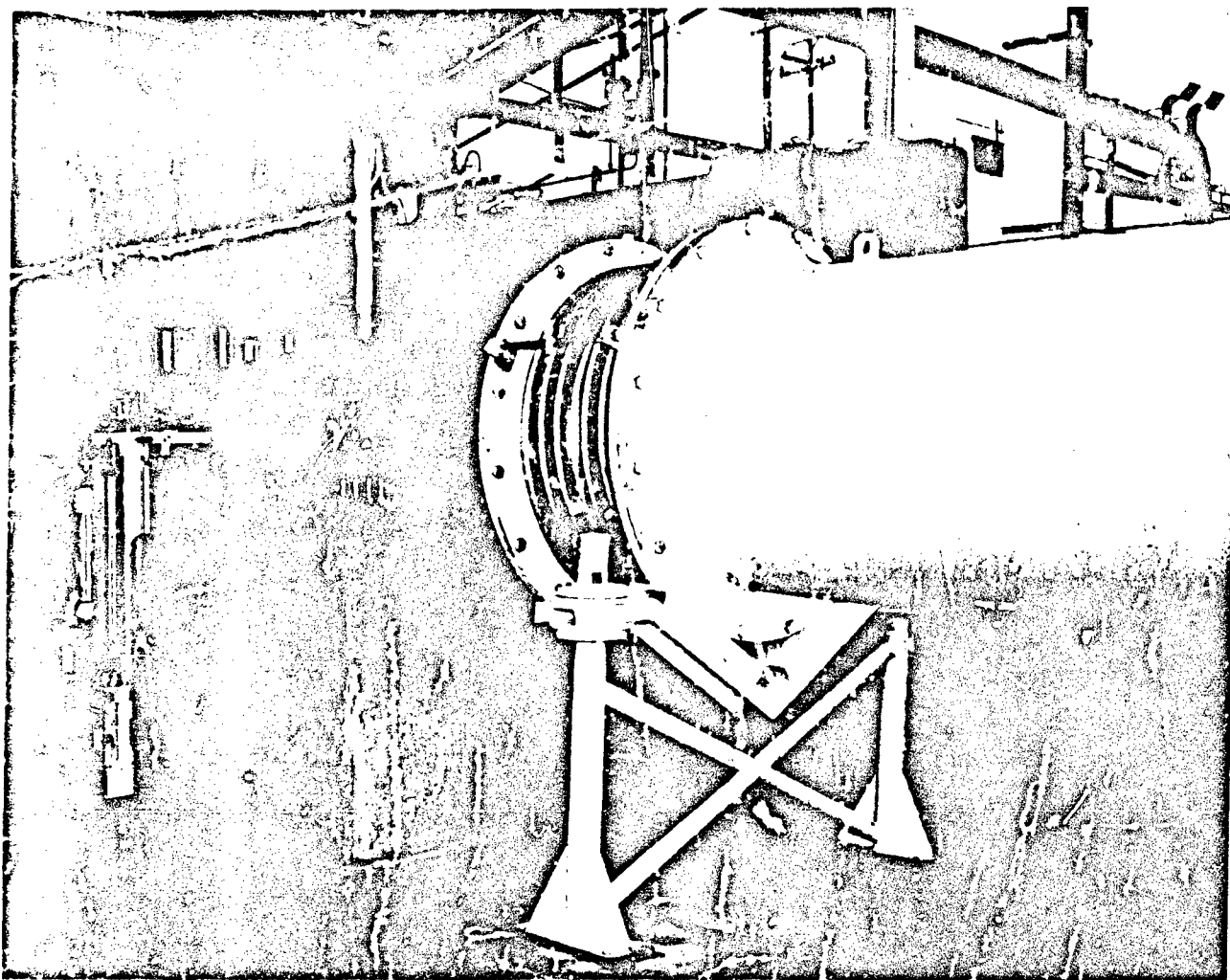
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Figure 35. Exhaust Duct

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Report AFMPL-TR-66-230



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Figure 36. Test Stand Enclosure and Bellows

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Report AFRPL-TR-66-230

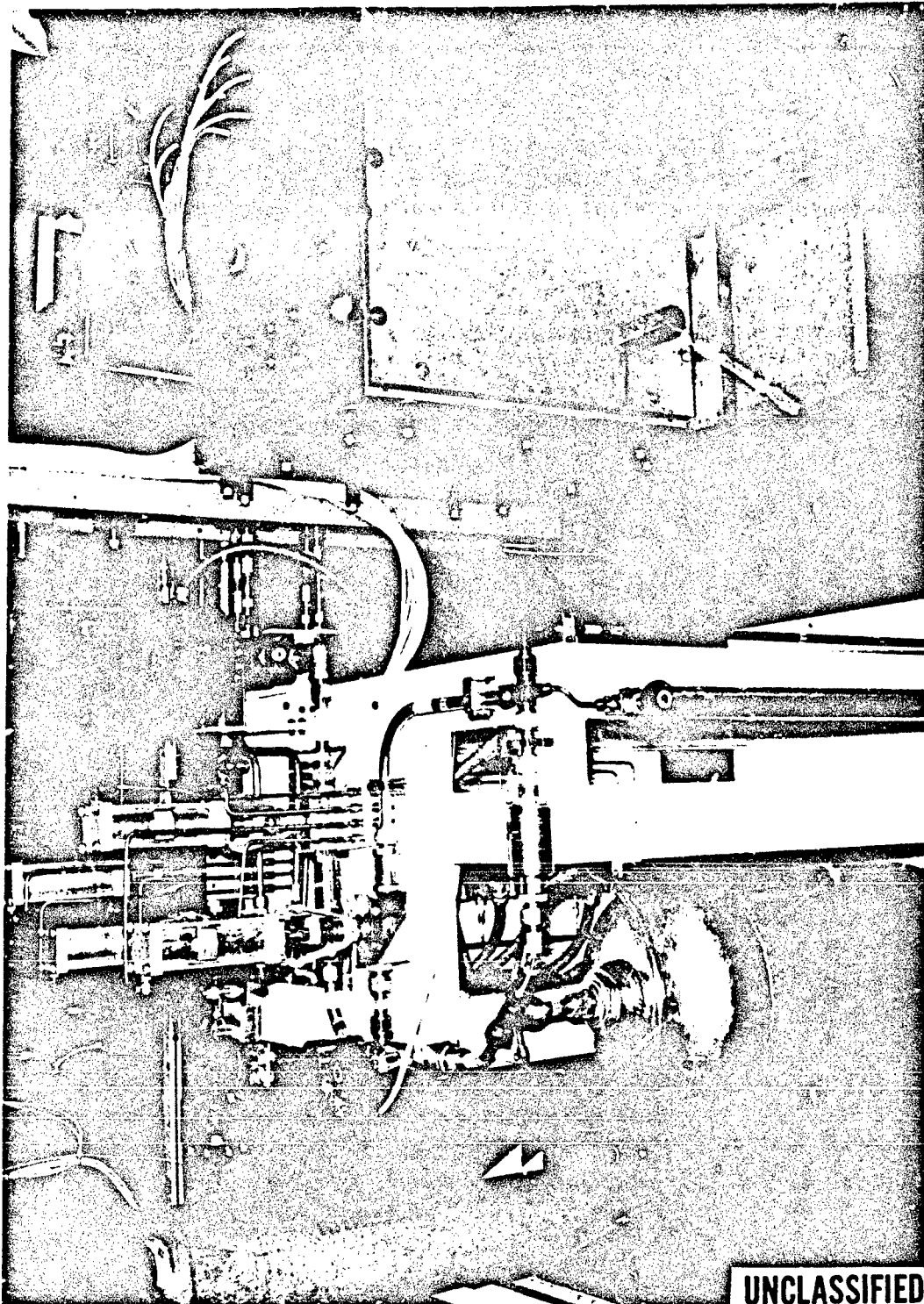


Figure 37. Oxidizer Feed System

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

Figure 36 depicts the feed-system components mounted on a steel framework. The oxidizer tank is located in the foreground adjacent to its sight glass. The other components control the flows of oxidizer and the nitrogen gas used for pressurization and injector purging. The mounting and arrangement of the components are of conventional design. Figure 38 is a schematic of the system.

(C) To the right of the oxidizer feed system, the rear of the vacuum enclosure may be seen. On top are the electrical connections for instrumentation. The rear plate of the enclosure is secured with springs permitting the entire plate to act as a relief valve in the event of an over-pressure. The hand valve on the side of the enclosure was opened after each firing to allow air inside the enclosure and return the system to ambient pressure.

#### (d) Fuel Feed System

(C) A side view of the fuel-feed system may be seen in Figure 39. The smaller tank contained fuel and the larger tank, behind the steel framework, contained the water used for positive displacement of the fuel. The Beryllizine and water were separated by a bladder of butyl rubber located in the fuel tank. A turbine-type flowmeter was located in the water line connected between the two tanks. During testing, the rate of water flow was used in conjunction with the measured density of the fuel for computation of fuel weight flow. Positive displacement for fuel feed eliminated the problems usually encountered with the direct measurement of flow rates of non-Newtonian liquids (e.g., flowmeter accuracy and repeatability). The mounting and arrangement of the other feed-system components are of conventional design. A schematic of the system may be found in Figure 40.











#### (e) Thrust Chamber Valves

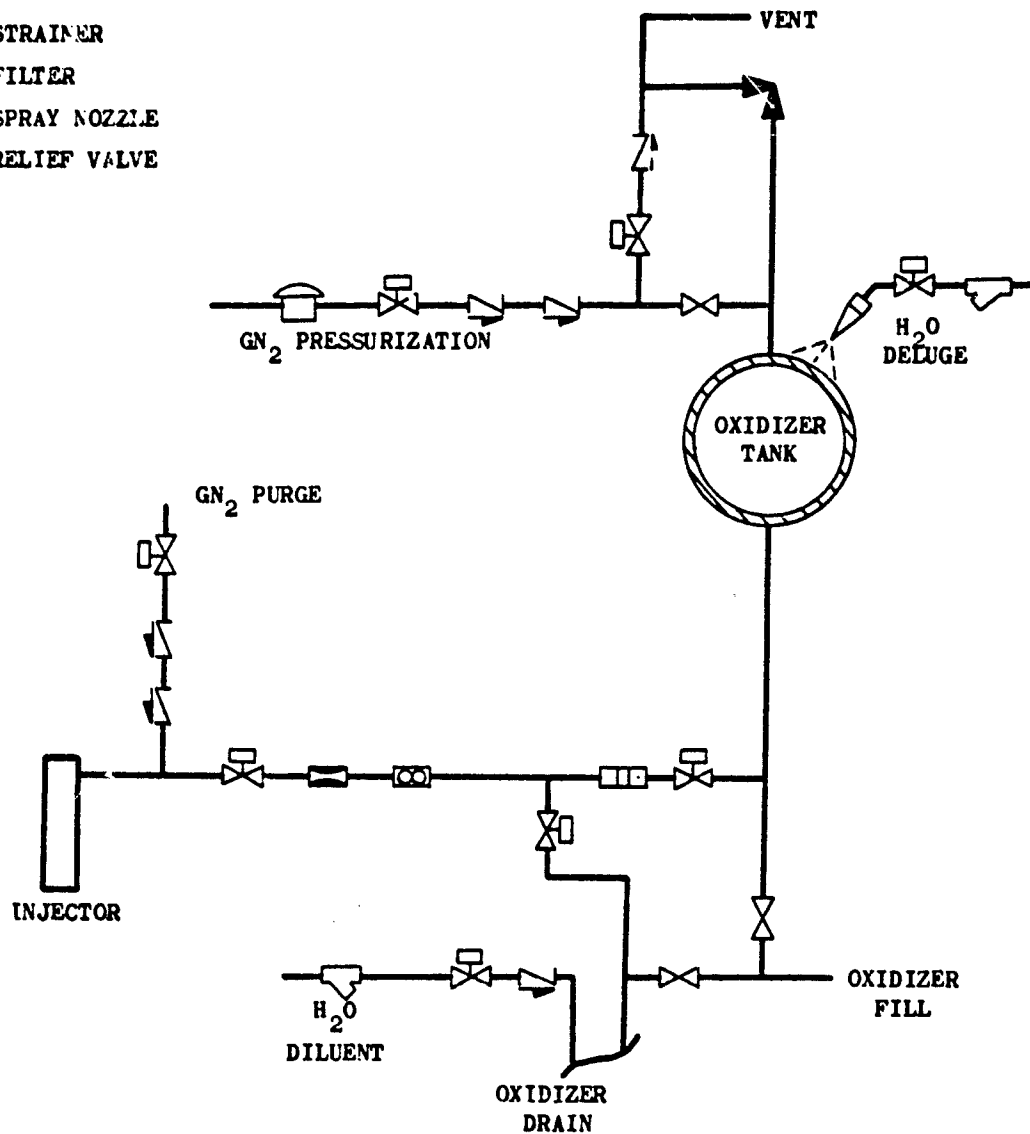
(C) Figure 41 shows the method of mounting and the arrangement of the thrust chamber valves and the safety valve in the fuel system. Between the fuel valves a tee may be seen. The leg of the tee supplied fuel to the flow loop which was used in another portion of the program. Located on top of the actuators for the thrust chamber valves were potentiometers. The potentiometers were wired as voltage divider and the shafts were rigidly affixed to the valve stems. The potentiometers were energized with voltage and their outputs were fed to an oscillograph. This feature permitted both the position and the behavior of the valves to be monitored during testing.

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Report AFRPL-TR-66-230

SYMBOLS

-  -- PRESSURE REGULATOR
-  -- REMOTE VALVE
-  -- HAND VALVE
-  -- CHECK VALVE
-  -- VENTURI
-  -- FLOWMETER
-  -- STRAINER
-  -- FILTER
-  -- SPRAY NOZZLE
-  -- RELIEF VALVE



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Figure 33. Schematic, Oxidizer Feed System

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Report AFRPL-TR-65-230

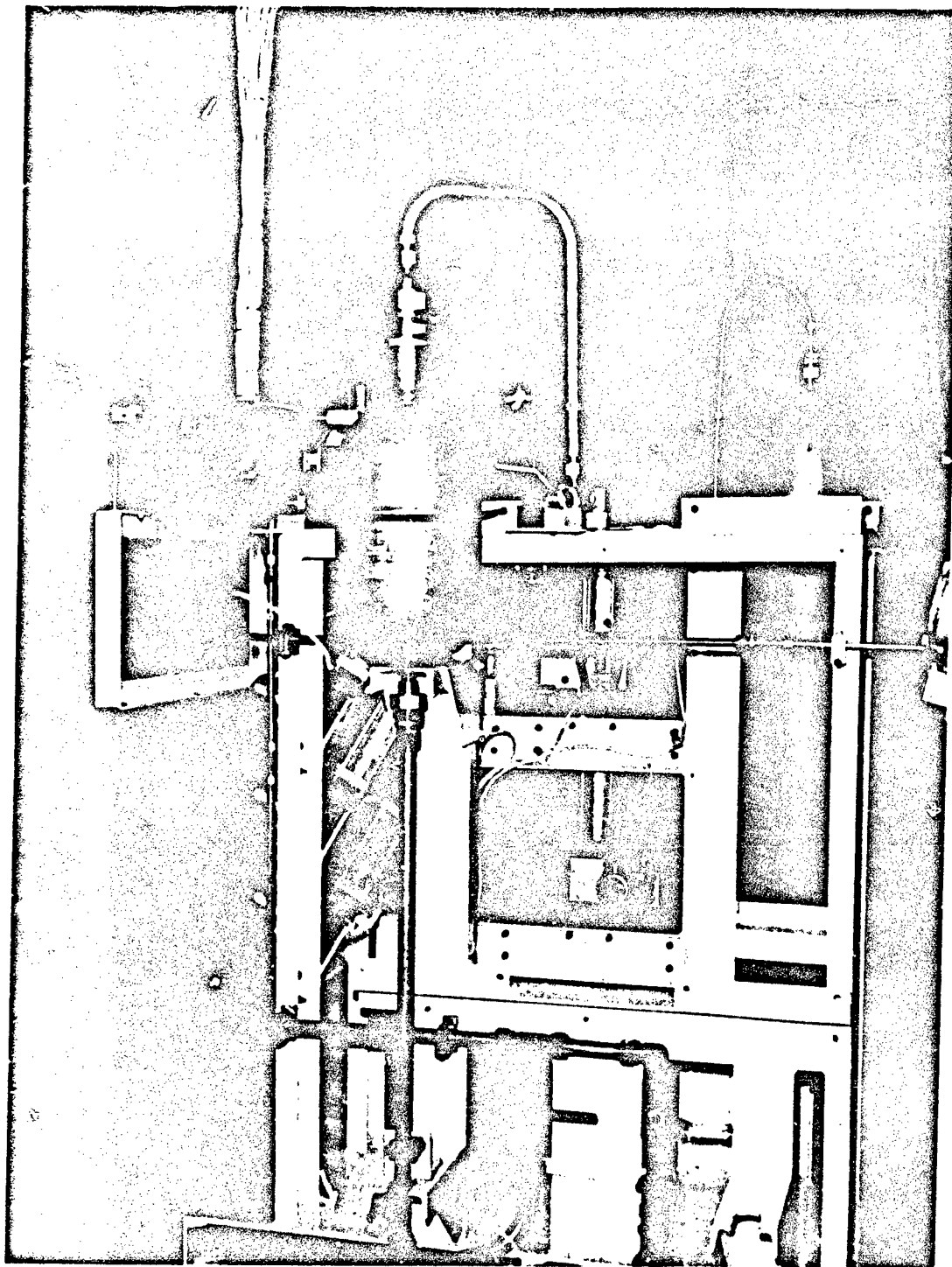


Figure 39. Fuel Feed System

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Report AFRPL-TR-66-230

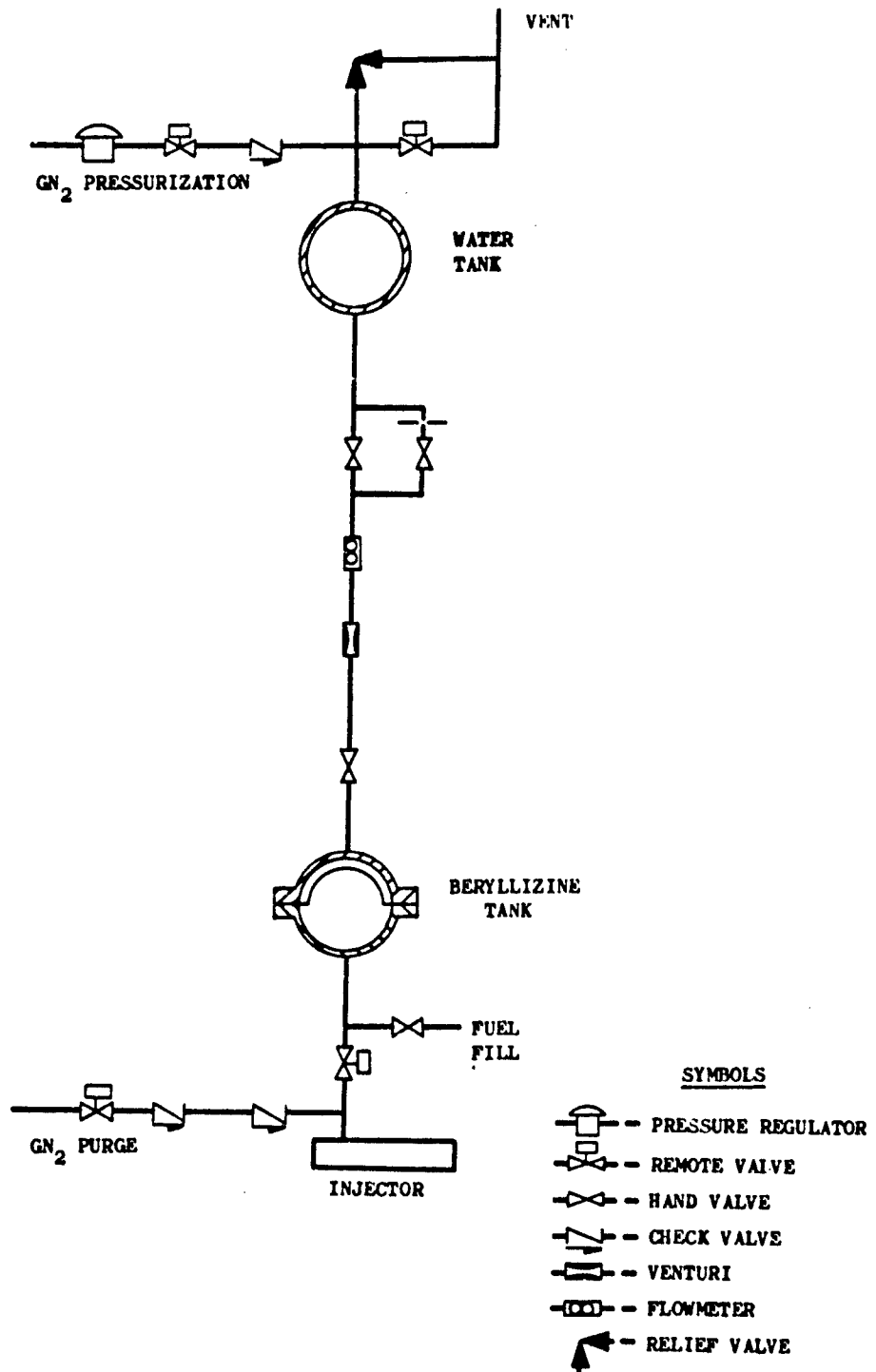


Figure 40. Schematic, Fuel Feed System

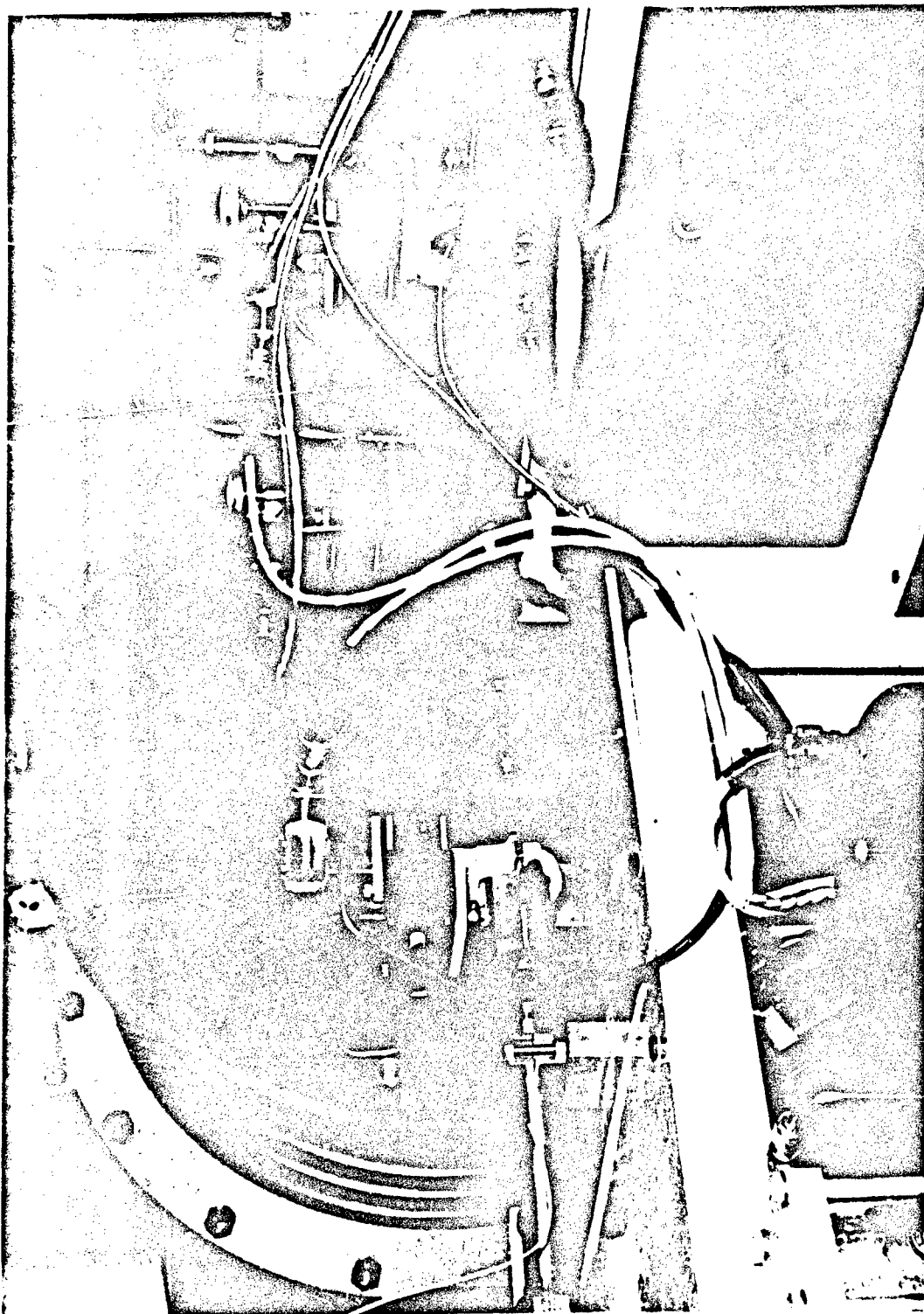
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Report AFRPL-TR-66-230



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Figure 41. Thrust Chamber Valves

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(C) A snap switch may be seen in position on the oxidizer valve. This switch was normally open until actuated and controlled the opening and closing signal to the fuel valve. It was operated by a cam on the stem of the oxidizer valve and was actuated when the oxidizer valve was 25% open. This switch assured that the fuel valve got no signal until the oxidizer valve was opening and closed the fuel valve in the event of premature closing of the oxidizer valve.

(C) Located behind the valves is the manifold for feeding the nitrogen gas used for injector purging. The nitrogen gas was fed from a common tube (upper right of the picture) into a tee. Check valves were located in the leg and run of the tee. A length of tubing connected the outlet of those check valves to the inlets of two check valves located at the outlets of the fuel and oxidizer thrust chamber valves. A small hole, 1/32 in. dia, was drilled in the tubing which connected the check valves. The nitrogen pressure was 150 psig and the gas was turned on before a test and was left on throughout the test. Should a primary check valve have leaked backwards during a test, the fuel or oxidizer would escape from the small hole drilled in the tubing connecting the pairs of check valves, indicating the check valve is leaking and should be replaced.

(C) The hand valves used for low-point bleeds can be seen behind the fuel and oxidizer valves. The valves used for high-point bleeds are located closer to the fuel and oxidizer tanks.

#### (2) $N_2O_4$ /Beryllizine-21 Ignition Testing

(C) Twenty-five tests were conducted with the  $N_2O_4$ /Beryllizine-21 propellant system. Twenty-three tests were conducted with the prototype fuel (Beryllizine made with the Brush Beryllium Company FP 1755 beryllium powder, hydrazine, and Carbopol as the gelling agent), and the remaining two tests were performed with beryllium powder of small diameter (Brush Beryllium Company 555 beryllium powder) and chromium-coated beryllium powder (Berylco product) in the Beryllizine, respectively. An additional test was conducted with the  $N_2O_4$ /Alumizine system for calibration purposes, bringing the total number of tests to 26. At the conclusion of the successful test series, the Centrojet injector was selected for the balance of the tests with  $N_2O_4$ /Beryllizine-21. Because ignition testing with  $N_2O_4$ /Beryllizine-21 was completed, the ignition tests with 98%  $H_2O_2$ /Beryllizine-28 were begun.

(C) In all but two ignition tests with  $N_2O_4$ /Beryllizine-21, the beryllium powder was ignited by the hot-core section of the injector. The test data and the analyzed results for all tests can be found in Tables XXXVII and XXXVIII, and Figures 42, 43, and 44.

CONFIDENTIAL

Report AFRPL-TR-66-230

TABLE XXXVII

$N_2O_4$ /BERYLIZINE-21 TEST RESULTS (U)

Test No.	Duration of Pull Pcs. sec	Chamber Pressure, Puls (1)	Ambient Pressure, Puls (2)	Thrust F. lb.	Fuel Flow Rate, lb/sec (3)	Oxidizer Flow Rate, lb/sec	Mixture Ratio	Total Flow Rate, lb/sec	Throat Dia. Prefire in.	Throat Dia. Postfire in.	Nozzle Exit Dia. in.	Area Ratio	Beryllizine Batch No. (4)
1K-2B-101	2.160	879.60	0.476	935.50	2.019	1.246	0.617	3.265	0.915	0.918	2.995	10.65	31P-3
1K-2B-102	2.250	815.35	1.906	889.20	2.007	1.201	0.598	3.208	0.905	0.939	2.991	10.15	31P-3
1K-2B-103	1.950	792.20	0.711	879.10	2.032	1.183	0.582	3.215	0.939	0.941	3.090	10.22	31P-3
1K-2B-104	2.0	745.7	0.446	771.5	2.147	1.266	0.590	3.413	0.910	0.939	3.033	10.71	31P-3
1K-2B-105	1.75	527.1	1.445	567.8	2.149	1.237	0.576	3.386	0.939	0.931	3.034	10.56	31P-6
1K-2B-106	2.11	763.1	0.279	883.5	2.140	1.247	0.582	3.387	0.931	1.003	3.023	10.02	31P-6
1K-2B-107	2.22	700.2	0.356	914.7	2.137	1.262	0.591	3.399	1.003	1.041	3.025	8.84	31P-6
1K-2B-108	1.8	505.1	0.467	638.2	2.170	1.245	0.574	3.415	1.041	1.035	3.041	8.56	31P-6
1K-2B-109	0.95	(784)	0.192	822.8	(2.133)	1.306	(0.612)	(3.439)	0.918	---	---	---	31P-7
1K-2B-110	1.95	(798)	0.354	852.7	(2.128)	1.303	(0.612)	(3.431)	---	0.978	3.036	10.10	31P-7
1K-2B-111	0.44	706.6	0.53	958.0	2.216	1.359	0.599	3.545	0.978	0.975	3.050	9.67	31P-7
1K-2B-112	1.9	508.9	0.256	596.3	2.152	1.338	0.622	3.490	0.975	0.987	3.050	9.70	31P-8
1K-2B-113	1.97	913.6	0.220	990.0	2.162	1.347	0.623	3.509	0.913	0.946	3.023	10.59	31P-8
1K-2B-114	0.47	891.4	0.208	957.0	2.154	1.393	0.647	3.547	0.916	0.916	3.024	10.90	31P-8
1K-2B-115	1.98	976.3	(0.33)	1076.2	2.177	1.515	0.696	3.692	0.916	0.946	3.020	10.60	31P-9
1K-2B-116	2.06	938.9	(0.15-0.77)	1006.0	2.154	1.525	0.708	3.679	0.916	0.948	2.996	10.43	31P-10
1K-2B-117	2.04	730.2	(0.50)	827.6	2.162	1.254	0.580	3.416	0.913	1.187	3.026	10.1	31P-10
1K-2B-118	2.09	918.4	(0.52)	978.0	2.158	1.554	0.720	3.712	0.934	1.000	3.017	10.34	31P-11
1K-2B-119	2.00	975.6	0.195	1061.9	2.156	1.567	0.727	3.723	0.912	1.017	3.028	10.55	31P-11
1K-2B-120	2.0	891.0	0.222	961.8	---	1.582	---	---	0.912	0.983	3.036	10.40	31P-11
1K-2B-121	1.64	1060.6	0.418	1094.5	2.229	1.577	0.699	3.786	0.922	0.955	3.035	10.62	31P-11
1K-2B-122	2.04	1101.8	0.504	1168.1	2.233	1.581	0.708	3.814	0.912	0.977	3.036	11.03	31P-12
1K-2B-123	2.04	1102.2	2.008	1211.1	2.226	1.551	0.697	3.777	0.914	0.935	5.645	37.94	31P-12
1K-2B-124	1.98	1019.8	0.262	1168.2	2.258	1.580	0.700	3.838	0.912	0.914	5.687	39.0	31P-13 (Al-430)
1K-2B-125	2.04	1023.7	0.297	1168.2	2.132	1.541	0.703	3.733	0.912	0.951	5.687	37.6	31P-14 (Sm Dia Be)
1K-2B-126	1.31	(880)	0.400	1012.8	2.224	1.486	0.668	3.710	0.918	0.921	5.687	38.2	31P-15 (Cr Coated Be)

NOTES:

- (1) Chamber pressure is the average value of two measurements taken from the same plenum port.
- (2) Ambient pressure is measured at the top of the vacuum enclosure which surrounds the test stand.
- (3) Fuel rate of flow is measured by positive displacement with water.
- (4) Propellant properties may be found in the chemistry section of this report.

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Report AFRPL-TR-66-230

TABLE XXXVIII  
N<sub>2</sub>O<sub>4</sub>/BERYLLIUM-21 TEST RESULTS (U)

Test No.	Time, sec (1)	Specific Impulse, sec		Theoretical Velocity, ft/sec		% of Theoretical		Thrust Coefficient		% of Theoretical	Posttest Remarks
		Theoretical	Experimental	Theoretical	Experimental	Theoretical	Experimental	Theoretical	Experimental		
11-2B-101	2.00	353.74	286.5	6669	5732	80.99	91.44	1.8153	1.6080	88.58	Beryllium ignition, stable test, heavy face erosion.
11-2B-102	2.00	349.40	271.2	6273	5658	79.34	90.20	1.7918	1.5760	87.96	Beryllium ignition, stable test, mild face erosion.
11-2B-103	2.00	352.83	273.4	6279	5514	77.49	87.82	1.8077	1.5955	88.26	Beryllium ignition, stable test, no face erosion.
11-2B-104	2.00	354.42	226.05	6273	4696	63.78	74.87	1.8177	1.548	85.6	Swirlor blew out on start, injector face burnt through.
11-2B-105	2.95	349.54	167.70	6263	3428	47.98	54.73	1.7953	1.574	87.67	No beryllium ignition, impingement point far from face.
11-2B-106	2.00	353.32	260.86	6277	5219	73.83	83.15	1.8108	1.608	88.80	Beryllium ignited, hardware undamaged.
11-2B-107	2.00	347.84	269.15	6269	5384	77.38	85.88	1.7848	1.608	90.09	Beryllium ignited, hardware undamaged.
11-2B-108	2.00	346.89	186.9	6269	4657	53.88	74.29	1.7802	1.291	72.52	Swirlor burnt out during run.
11-2B-109	1.8	354.33	239.2	6265	4984	67.51	79.55	1.8194	1.544	84.86	Short run, lost P <sub>0</sub> ; fuel leakage noted; some instability.
11-2B-110	2.00	352.45	257.2	6263	5094	72.98	81.33	1.8102	1.569	86.58	P <sub>0</sub> tap plugged; fuel leakage noted; injector slightly burnt.
11-2B-111	1.25	350.50	242.0	6266	4812	69.04	76.80	1.7995	1.618	89.91	Purges did not function; no beryllium ignition.
11-2B-112	2.00	349.78	170.8	6241	3534	48.83	56.63	1.8029	1.556	86.30	
11-2B-113	2.00	354.03	282.2	6268	5647	79.71	90.10	1.8171	1.609	88.55	
11-2B-114	1.35	374.28	269.8	6256	5329	76.15	85.18	1.8217	1.659	89.42	
11-2B-115	2.00	351.76	291.5	6239	5760	82.87	92.32	1.8137	1.628	89.76	Short run; fuel supply depleted.
11-2B-116	2.00	350.19	273.4	6232	5573	78.07	89.43	1.8078	1.579	87.35	
11-2B-117	1.5	352.99	242.3	6276	4884	68.64	77.82	1.8094	1.596	88.21	Oxidizer venturi partially plugged; anilative throat badly eroded.
11-2B-118	2.00	349.96	263.5	6225	5477	75.29	87.98	1.8084	1.548	86.60	
11-2B-119	2.00	350.94	285.2	6225	5756	81.27	92.46	1.8135	1.594	87.90	Small fuel leak (correction applied).
11-2B-120	Not analyzed.										Burnt transducer line; lost fuel flow rate.
11-2B-121	2.00	351.71	289.05	6242	6116	82.18	97.99	1.8127	1.521	83.91	
11-2B-122	2.00	352.51	306.27	6240	6101	86.88	97.78	1.8174	1.615	88.86	
11-2B-123	2.00	371.66	320.63	6244	6193	86.27	99.18	1.9147	1.666	87.01	Nozzle extension and extra chamber section added.
11-2B-124	2.00	356.91	304.36	5957	5592	85.27	95.53	1.9616	1.751	89.26	As above, AL-430 used as fuel.
11-2B-125	2.00	382.18	312.93	6238	5961	81.86	95.56	1.9708	1.689	89.70	As in 11-2B-123, porous injector face, small leak used in fuel.
11-2B-126	Not analyzed as of reporting date.										Chromium coated Be used in this test.

(1) The time when the data were analyzed corresponds to the time from fire-switch.

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Report AFRPL-TR-66-230

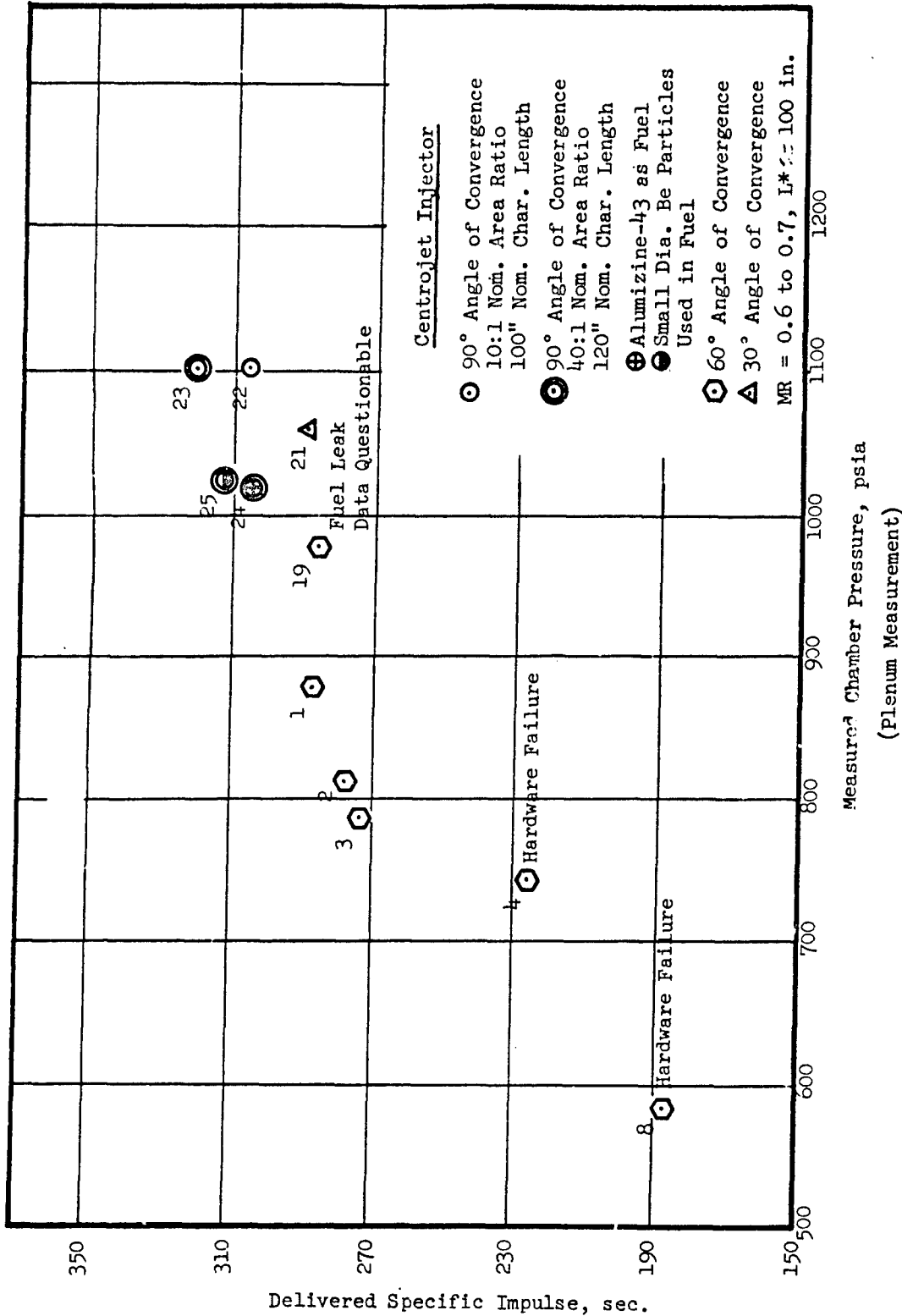


Figure 42.  $N_2O_4$ /Beryllizine-21 Tests, Centrojett Injector (U)

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Report AFRPL-TR-66-230

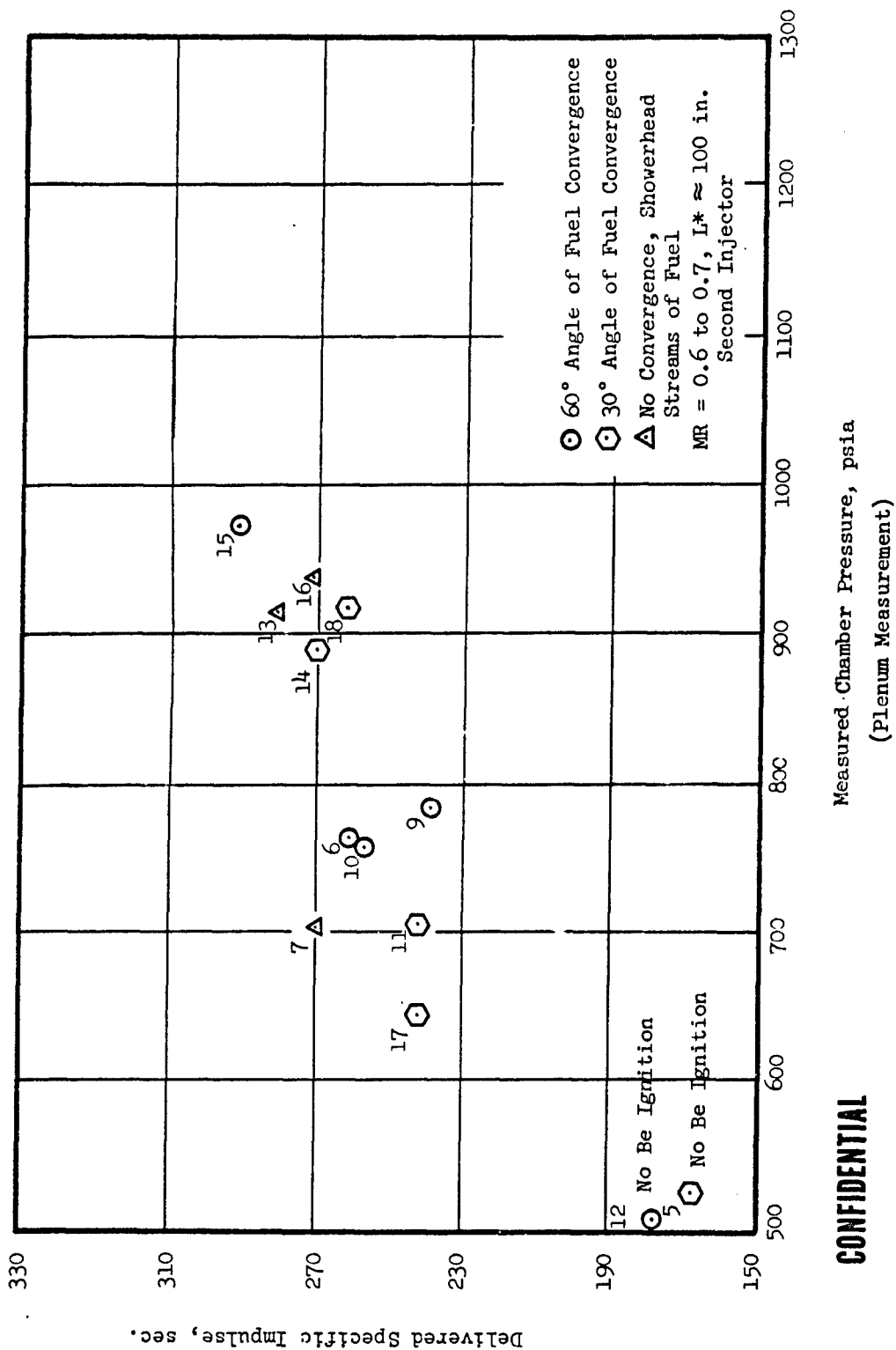


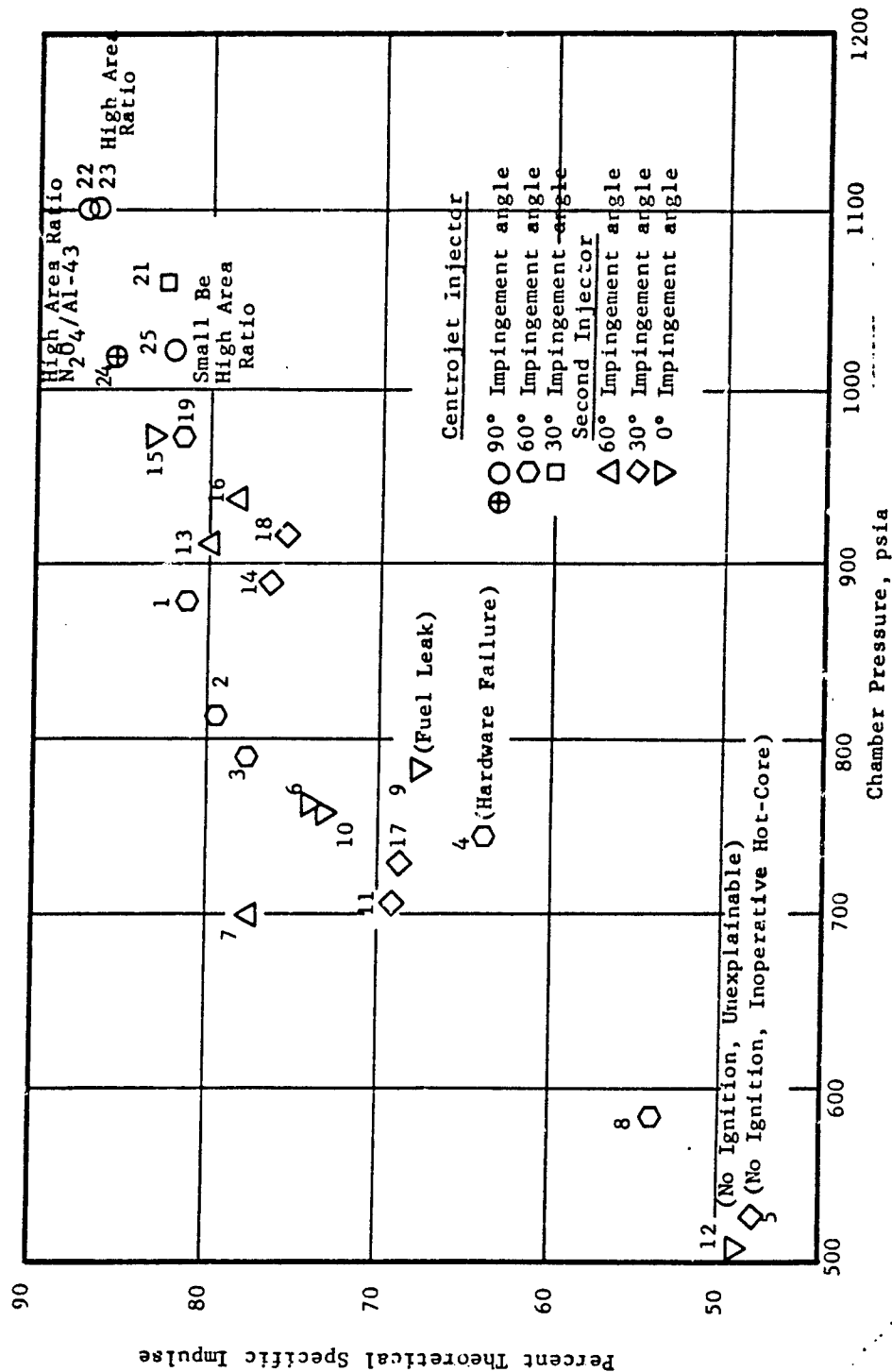
Figure 43.  $M_{2O_4}$ /Beryllizine-21 Tests, Second Injector (U)

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Figure 44. Experimental Performance of  $N_2O_4$ /Beryllizine-21 (U)

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) The following paragraphs discuss the pretest and post-test conditions and circumstances during each test.

#### Test 1k-2B-101

(U) The modified Centrojet injector, with the 60° deflector ring, was used for the first test. The combustion chamber was assembled using four chamber sections containing ablative inserts of phenolic material reinforced with Refrasil. The nozzle convergence and divergence were of the same ablative material, and the throat was fabricated from ATJ graphite.

(C) After the test, the hardware was examined for damage. The porous face was eroded and the fuel swirler used in the hot-core section of the injector was burned. The damage was caused by insufficient flow of oxidizer through the porous face because of oxidizer leakage at the margins of this component. The combustion chamber was eroded and contained deposits of a white, powdery substance. The volume of deposits increased with distance from the injector face. The nozzle convergence was badly eroded and the throat was streaked by particle erosion and discolored by heat. The nozzle divergence contained sporadic deposits of grey-white material. There was a hard coating, of approximately 1/16 in. thickness, on the downstream surface of the throat. Analysis by X-ray diffraction revealed the white powder was beryllium oxide.

#### Test 1k-2B-102

(U) The modified Centrojet injector was repaired and a new, porous face machined to accept a thread of zinc chromate paste around the periphery of the face and O-rings around the heads of the screws which hold the face in position. The nozzle and last section of the combustion chamber were replaced for this test.

(U) After the test, it was found that the thread of zinc chromate paste had stopped all leakage around the periphery of the face and the O-rings had prevented leakage past the screws. The porous face was again eroded around the fuel swirler which was burned. The effects of combustion and erosion to the chamber and nozzle were similar to the previous test.

#### Test 1k-2B-103

(U) Prior to this test, the burned swirler was replaced and its length was increased by 1/8 inch and threaded. The porous face was grooved around the swirler element to accept an O-ring. A specially made nut was used to hold the O-ring in its groove and to retain the center portion of the porous face in position. The thrust chamber from the previous firing was used for this test.



## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) After the test, the porous face was intact and uneroded except for a burned spot of approximately 1/16 in. dia. This type of burning was similar in appearance to that which occurs during shutdown if there is insufficient oxidizer flowing through the porous face to prevent recirculation of the burning particles. A longer oxidizer lag at shutdown was planned to prevent further burning and the electrical sequencer, used to control the firings, was set accordingly. There was no other damage to the injector; it was prepared for refiring with no further modifications. The combustion chamber sustained mild erosion and was acceptable for an additional firing, but the nozzle was heavily eroded in the convergent section and needed refurbishment prior to the next test.

#### Test 1k-2B-104

(U) The modified Centrojet injector with the 60° deflector ring was again used for this test. The combustion chamber from the previous test was combined with a new nozzle. This was to be a repeat firing of the previous test.

(C) After the test, the porous face was found to be burned through at a point corresponding to the spot of erosion sustained during the previous test. The porous face was eroded around the burnthrough and the central hole. The swirler and the specially made nut to hold the face in position were missing. Review of the oscillogram revealed that the swirler was damaged during the start transient. The oxidizer then flowed from the central hole in the face instead of through the porous face throughout the test. The porous face burned through just prior to shutdown as evidenced by the oscillogram. The thrust chamber was noticeably damaged. Even though the hot-core was inoperative after the start transient, the chamber pressure did not recede, indicating the beryllium burned throughout the test.

#### Test 1k-2B-105

(U) The second injector was mounted on the test stand with the thrust chamber from the previous test. The injector insert used for this test converged the streams of fuel at 30°.

(C) After the test, the chamber was found to be filled with Beryllizine nearly to the level of the throat. Further examination revealed neither hardware damage nor evidence of ignition of the beryllium powder. The failure of the beryllium to ignite was subsequently traced to the inoperative hot-core. The point of impingement of the streams of oxidizer on the stream of fuel was too far from the injector face. The oxidizer streams lacked sufficient momentum to penetrate the central fuel stream and ignite the beryllium particles.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### Test 1k-2B-106

(U) The second injector, together with the insert which converges the fuel at 60°, was mounted on the test stand. The combustion chamber and nozzle were used in the previous test.

(U) Subsequent to firing, the injector face was found to be clean and free from any damage or erosion. The combustion chamber and nozzle were found to be relatively undamaged.

#### Test 1k-2B-107

(U) The second injector, along with the insert which injects streams of fuel in a showerhead configuration, was used for this test. The chamber and nozzle were used in the previous test.

(U) Post-test examination revealed the injector face to be clean and free from erosion. The convergent section of the nozzle was heavily eroded and the throat had appreciably increased in diameter. The combustion chamber liners were eroded and not considered to be in refireable condition.

#### Test 1k-2B-108

(U) The modified Centrojett with the swirler repaired and a new face-retaining nut was used for this test. The injector, along with a completely refurbished thrust chamber, was mounted on the test stand. The 60° deflector ring was installed for this test.

(U) Post-test examination of the hardware revealed the face retaining nut was missing, the swirler burned and erosion on the porous face. The internal surfaces of the thrust chamber and vacuum enclosure were coated with the white powder, which was previously analyzed as beryllium oxide.

#### Test 1k-2B-109

(U) The thrust chamber was fitted with a new throat insert for this test. The second injector with the insert which converges the fuel at the maximum angle, 60°, was mounted on the test stand.

(U) Post-test examinations revealed fuel leakage during the test from the fitting at the injector to feed line interface. The oscillogram showed that chamber pressure was not recorded during the test. The problem was subsequently traced to misalignment of the hole drilled through the ablative insert in the combustion chamber from the boss for measurement of the pressure. The fuel fitting was tightened and the chamber pressure tap redrilled in preparation for the next test.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### Test 1k-2B-110

(U) This test was a repeat firing of Test 1k-2B-109. Post-test examinations revealed shutdown-type erosion on the face of the injector, but it was refireable. The combustion chamber and nozzle received damage considered to be normal.

#### Test 1k-2B-111

(U) The thrust chamber from the previous test was refired along with the second injector. The injector insert selected for this test converged the streams of fuel at 30°, and was a repeat of Test 1k-2B-105. Two injector modifications were performed prior to this test. The oxidizer orifices for the hot-core section of the injector were welded closed and redrilled for impingement closer to the injector face; additionally, the fuel injection elements were welded to the face of the injector to eliminate leakage of oxidizer from the threads.

(C) Post-test investigations revealed that (1) the hot-core had functioned properly since the beryllium had ignited, (2) shutdown-type erosion on the injector face, and (3) normal erosion of thrust chamber components.

#### Test 1k-2B-112

(U) The thrust chamber from the previous test was mounted on the test stand. The injector for this test was the second design with the insert which converges the fuel at 60°.

(C) Post-test examinations revealed neither ignition of the beryllium nor damage to the test hardware. The malfunction of the hot-core cannot be explained.

#### Test 1k-2B-113

(U) A new thrust chamber and the second injector with the insert containing showerhead injection elements for fuel was used for this test.

(C) Post-test examinations revealed that the beryllium powder had ignited, the injector face was neither burned nor eroded, and the interior surfaces of the thrust chamber were extremely clean and relatively undamaged.

#### Test 1k-2B-114

(U) This test was a repeat firing of Test 1k-2B-111. The injector used was the second design with the fuel converging at 30°. A refurbished thrust chamber was used for this test.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(C) Post-test examinations revealed the beryllium powder had ignited and the thrust chamber received mild damage. The injector face was eroded slightly during shutdown.

#### Test 1k-2B-115

(U) This test was the last firing with the insert for the second injector which converges the fuel at 60°. The thrust chamber from the previous test was used.

(U) Post-test examination showed an eroded injector face, caused by the recirculation of burning metal particles; however, the unit was refireable.

#### Test 1k-2B-116

(U) This test was a repeat firing of Test 1k-2B-113 with an increased rate of oxidizer flow. The second injector, with the fuel showerhead insert, was used. The thrust chamber from the previous test was refurbished with a new throat and used for this test.

(C) Post-test examination of both the data and test hardware revealed successful ignition of the beryllium powder, slight erosion to the face of the injector, and mild discoloration and erosion of the combustion chamber inserts.

#### Test 1k-2B-117

(U) A thrust chamber refurbished with new inserts was used for this test. The injector was the second design with the insert that converges the streams of fuel at 30°.

(U) Post-test examinations revealed a lower-than-anticipated mixture ratio caused by a low rate of oxidizer. The cavitating venturi used to control the rate of oxidizer flow was found partially blocked by a piece of Teflon. The material came from the oxidizer thrust chamber valve and was apparently sheared from the seat when the valve opened. The seats and seals were replaced prior to the next test.

#### Test 1k-2B-118

(C) This test was a repeat firing of the previous test. After the test the injector, combustion chamber and nozzle were found in excellent condition. Review of the data showed the beryllium had ignited.

#### Test 1k-2B-119

(U) The Centrojet injector with the 60° deflector ring was used for this test. The thrust chamber from the previous test was refurbished with a new throat in preparation for this test.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) After the test, the injector face was severely eroded. Review of the data revealed an abnormally high differential pressure across the porous face, indicating low porosity and inadequate cooling. The combustion chamber was found to be eroded slightly.

#### Test 1k-2B-120

(U) The Centrojet injector was used for this test, along with the 30° deflector ring. The thrust chamber from the previous test was fitted with a new throat. The rate of oxidizer flow to the porous face of the injector was increased in an attempt to prevent face erosion.

(U) Post-test examination revealed an eroded injector face; however, the erosion was less severe in the peripheral area than was encountered in the previous test. The erosive damage beneath the hot-core pattern remained the same. The combustion chamber inserts were damaged slightly, but the nozzle was not in refireable condition. Further investigation revealed the fuel-manifold transducer for pressure measurement had burst during the start transient. The resultant fuel leak was quite large throughout the test, so performance information was unobtainable for this test (the transducer was later found to have been defective).

#### Test 1k-2B-121

(U) This test was a repeat firing of the previous test using the same test hardware.

(U) After the test, the injector face was damaged slightly more than the one used for the previous test. The combustion chamber and nozzle were found to be in normal post-test condition.

#### Test 1k-2B-122

(U) The Centrojet injector using the 90° propellant-deflector ring, along with a refurbished thrust chamber, was used for this test.

(C) The injector face was found heavily damaged after the firing. The combustion chamber inserts were found mildly eroded, and the convergent section of the nozzle was found damaged slightly in comparison with the results from other tests. The divergent section of the nozzle was found to be undamaged. The highest specific impulse (306.3 sec) and chamber pressure (1102 psia) obtained thus far were achieved during this firing; moreover, damage to the combustion chamber liner was approximately the same as that sustained during tests with other injectors, but nozzle damage was significantly less severe than sustained before. On the basis of these considerations the Centrojet injector using the 90° deflector ring was chosen for the balance of testing with  $N_2O_4$ /Beryllizine-21.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### Test 1k-2B-123

(C) Two significant changes were made to the test hardware prior to this test. The nozzle was modified to accept a skirt of high area ratio (40:1), and the length of the combustion chamber was increased by approximately 20% (one chamber section).

(C) Subsequent to firing, the injector face was found eroded beneath the impinging sheets of propellant from the hot-core section of the injector. The swirler and heads of the retaining screws were burned. The chamber inserts and nozzle were damaged slightly.

#### Test 1k-2B-124

(C) The test conditions remained the same for this test, except that Alumizine-43 was used for fuel. This test was considered to be a calibration test for the research combustor. Performance efficiencies (% I) in excess of 90% have been demonstrated consistently in thrust chambers at 5000 and 15,000 lb thrust levels operating at chamber pressures of 500 and 1500 psia, respectively. It was postulated that losses due to two-phase flow in the nozzle were high with the small combustor and the performance efficiencies being obtained at the time were indeed reasonable; also, if the thrust chamber assembly were scaled to a larger size, the performance efficiency would be similar to that obtained with  $N_2O_4$ /Alumizine.

(C) Post-test examination revealed the injector face to be burned through in three places and the nozzle more heavily damaged than observed after a firing with  $N_2O_4$ /Beryllizine-21. There was no further damage.

#### Test 1k-2B-125

(C) This test was conducted with Beryllizine containing small diameter beryllium particles. The objective of this test was to investigate the effect of particle size on ignition and performance efficiency. The thrust chamber was refurbished with new inserts, and a new porous face was installed in the injector.

(U) After the test the injector face was found to be clean and undamaged, as were the combustion chamber and nozzle. There was no further damage.

#### Test 1k-2B-126

(C) This test concluded the series of ignition tests with the  $N_2O_4$ /Beryllizine-21 propellant system. The test was conducted at the experimental conditions of the previous test, only the Beryllizine contained beryllium

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

particles coated with chromium. The object of this test was to determine the effect of chromium-coated particles on ignition and performance efficiency.

(C) Post-test review of the data revealed that the beryllium had been ignited by the hot-core and burned throughout the test. The oscillogram showed that chamber pressure had not been recorded throughout the test. When the hardware was inspected the porous face was found discolored by heat but neither eroded nor burned. The combustion chamber liners and nozzle were relatively undamaged. The drilled hole for measurement of chamber pressure was found plugged which explains why chamber pressure was not recorded.

#### (3) 98% $H_2O_2$ /Beryllizine-28 Ignition Testing

(C) Eleven tests were conducted with the 98%  $H_2O_2$ /Beryllizine-28 propellant system; however, only seven of these tests resulted in valid test data. This series of ignition tests consisted of nine thrust chamber tests and two tests of the hot-core alone.

(C) The initial three of the nine thrust chamber tests did not produce valid data because of hard starts during the start transient which damaged the injector face, or improper operation of other equipment which prematurely terminated the test. Preliminary to the start transient of the fourth test, a detonation occurred in the combustion chamber which demolished the test equipment inside the vacuum enclosure. Subsequent to the explosion, the equipment was rebuilt and a new method of preventing hard starts using Alclo igniters was adopted. After a short group of three igniter tests to develop a reliable ignition sensor, the first thrust chamber test was successfully conducted and produced valid data.

(U) During a routine inspection of the test equipment preliminary to the fifth test, fatigue cracks were found around the periphery of the oxidizer tank. A new tank was installed and prepared for testing. During the preparatory activities for the fifth test, the oxidizer tank exploded and severely damaged the oxidizer feed system. Enough components remained undamaged to reconstruct the system and resume testing. The remaining four ignition and combustion tests using the complete thrust chamber assembly were conducted without incident and produced valid data.

(U) Even though the third thrust chamber test (1k-2B-129) was terminated prematurely by a safety shutdown device, beryllium oxide was found inside the combustion chamber and vacuum enclosure. The presence of oxide indicates ignition of the beryllium powder prior to shutdown and proper operation of the hot-core with 98%  $H_2O_2$  and Beryllizine-28. The test data and the analyzed results for all tests can be found in Table XXXIX and Figures 45 and 46.

**TABLE XXXIX**  
**99% N<sub>2</sub>O<sub>4</sub>/BERYLLIZINE-28 TEST CONDITIONS AND RESULTS (U)**

Test No.	Test Conditions										Test Results			
	Duration of Full P.C. sec	Chamber Pressure, P <sub>c</sub> , PSIA (1)	Ambient Pressure, P <sub>a</sub> , PSIA (2)	Thrust F, lb	Fuel Flow Rate, W <sub>f</sub> , lb/sec (3)	Oxidizer Flow Rate, W <sub>o</sub> , lb/sec	Mixture Ratio W <sub>f</sub> /W <sub>o</sub>	Total Flow Rate, W <sub>t</sub> , lb/sec	Throat Dia., D <sub>t</sub> , in	Posttest Throat Dia., D <sub>p</sub> , in	Nozzle Exit Dia., D <sub>e</sub> , in	Area Ratio, A <sub>e</sub> /A <sub>t</sub>	Beryllizine Batch No. (4)	
1K-28-132A	1.10	976.6	0.404	1155.4	1.596	1.766	1.107	3.362	0.916	0.918	5.70	38.73	32P-28	
1K-28-132B	1.10	1066.7	0.449	1216.0	1.608	1.854	1.160	3.462	0.916	0.918	5.70	38.73	32P-28	
1K-28-133	0.19	971.6	0.383	1086.0	2.033	1.854	0.652	3.359	0.918	0.924	5.70	38.73	32P-30	
1K-28-134	0.25	971.6	0.383	1136.5	2.033	1.398	0.681	3.431	0.916	0.914	5.70	38.73	32P-31	
1K-28-135	0.35	1021.2	0.277	1166.9	2.052	1.361	0.663	3.413	0.914	0.912	5.70	38.73	32P-31	
1K-28-136A	0.38	1022.4	0.230	1199.6	1.783	1.675	0.939	3.458	0.912	0.910	5.70	39.15	32P-31	
1K-28-136B	0.38	1021.4	0.250	1196.2	1.812	1.672	0.923	3.484	0.912	0.910	5.70	39.15	32P-31	
Test No.	Time, Sec (5)	Theoretical		I <sub>sp</sub> Eff, sec	Experimental		C <sub>p</sub> Eff, %	Theoretical C <sub>p</sub>	Experimental		C <sub>p</sub> Eff, %	Posttest Remarks		
		I <sub>sp</sub> , Sec	I <sub>sp</sub> , Sec		I <sub>sp</sub> , Sec	I <sub>sp</sub> , Sec			C <sub>p</sub> , %	C <sub>p</sub> , %				
1K-28-132A	0.750	380.75	380.75	90.27	343.70	6159	100.07	1.9902	1.7950	90.19	90.19	Data obtained early in test.		
1K-28-132B	0.950	379.21	379.21	82.61	351.20	6193	100.09	1.9873	1.8250	91.81	91.81	Data obtained late in test.		
1K-28-133	0.150	370.51	370.51	82.79	323.30	6134	96.46	1.9833	1.7020	85.82	85.82	No Or. orifices in hot-core.		
1K-28-134	0.150	391.28	391.28	83.13	329.30	6311	95.39	2.0011	1.7570	87.80	87.80	Or. orifices in hot-core.		
1K-28-135	0.150	391.91	391.91	84.80	341.90	6342	100.16	2.0013	1.7340	86.64	86.64	Long combustion chamber.		
1K-28-136A	0.150	366.27	366.27	89.81	346.90	6317	99.73	1.9988	1.8000	90.05	90.05	Data obtained early in test.		
1K-28-136B	0.250	366.52	366.52	88.97	343.90	6148	98.80	1.9981	1.8000	90.09	90.09	Data obtained late in test.		

**NOTES:**

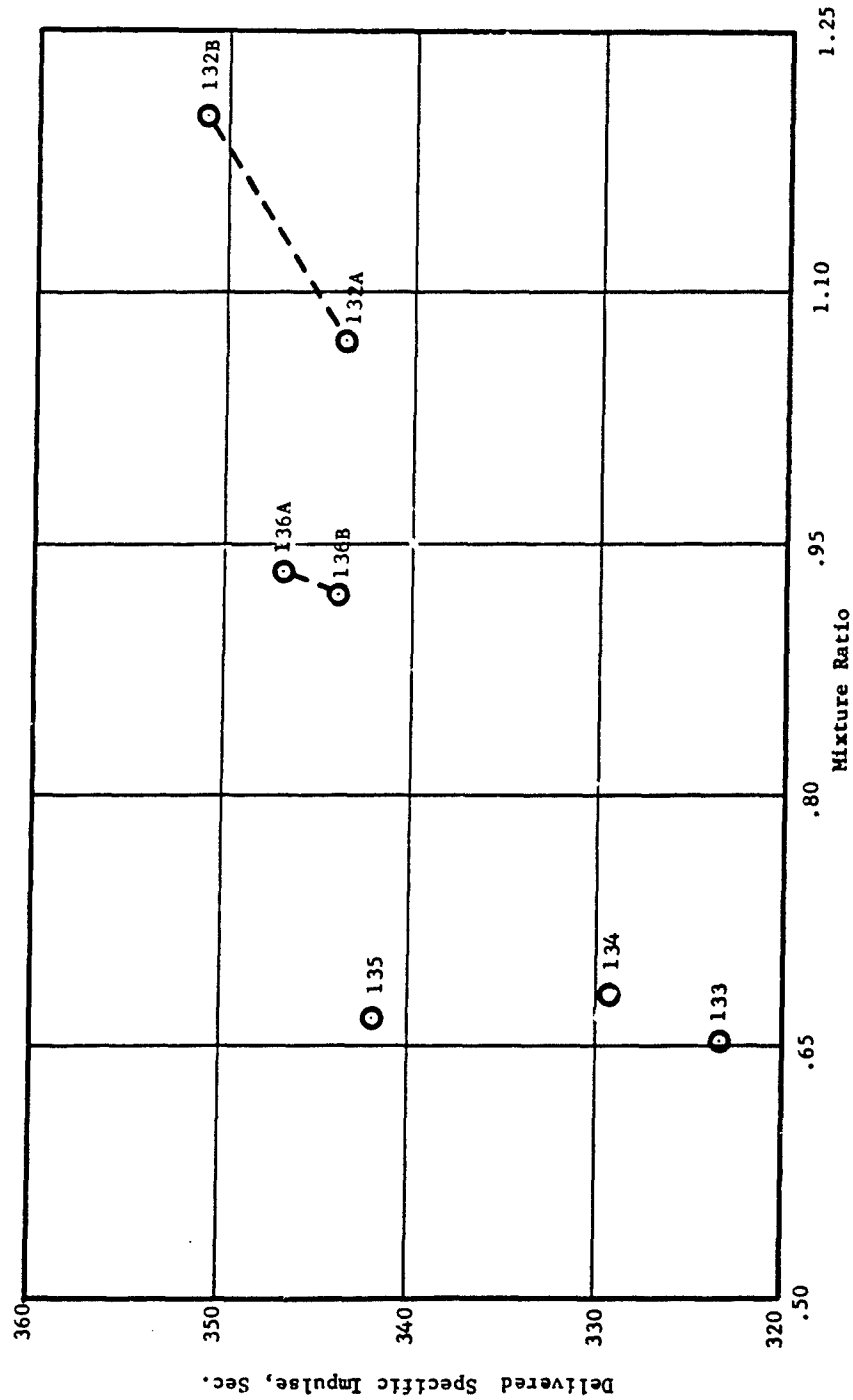
- (1) Chamber pressures are averages of two measurements.
- (2) Ambient pressure was measured inside the vacuum enclosure, above the TCA and on the plane of the nozzle exit.
- (3) Rate of fuel flow was obtained by measuring the flow rate of the pressurized water used for positive displacement.
- (4) Propellant properties are located in the chemistry section of this report.
- (5) Time of data reduction corresponds to the time from establishment of full chamber pressure.

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Report AFRL-TR-66-230



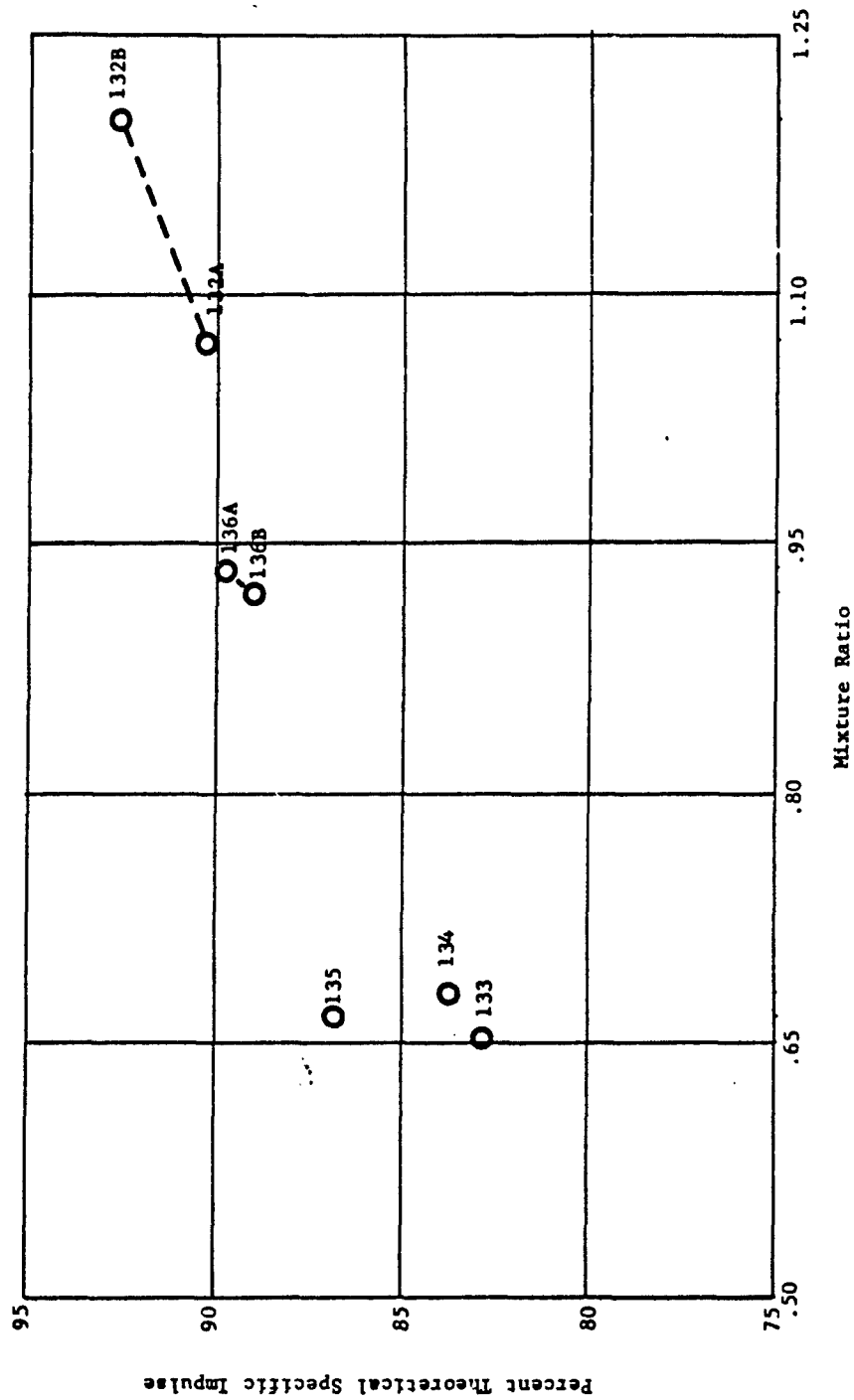
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Figure 45. 93%  $H_2O_2$ /Beryllizine-28 Tests, Centrojett Injector (U)

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Report AFRPL-TR-66-230



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Figure 46. Experimental Performance of 99%  $H_2O_2$ /Beryllizine-28 (U)

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Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) The following paragraphs discuss the test program with the 98%  $H_2O_2$ /Beryllizine-28 propellant system.

#### Test 1k-2B-127

(C) The Centrojet injector was not used for the first test because porous material for the transpiration cooled face was not available. Instead, the second injector with the insert which converges the peripheral streams of fuel at  $60^\circ$  was mounted on the test stand. The insert had been refurbished with a new porous face subsequent to its last test with  $N_2O_4$ /Beryllizine-21. The hydraulic design of the injector was not changed because of the similarity in fluid properties of  $N_2O_4$  and 98%  $H_2O_2$ , and Beryllizine-21 and Beryllizine-28. The standard length chamber (12.5 in., measured from the injector face to the nozzle entrance) along with the 40:1 nozzle extension was used for this test. The test was to be conducted at an approximate mixture ratio of 0.6 for 2 sec nominal duration.

(U) During the start transient of the test the hydrogen peroxide feed line burst just downstream of the thrust chamber valve. The overpressure was probably caused by contaminants in the oxidizer thrust chamber valve, since the seats were scorched and the interior surfaces of the valve were discolored by heat. Neither the source nor the identity of the contaminants is known. After repairing the damage, the oxidizer feed system was flowed with hydrogen peroxide at the design pressure to assure complete passivation. The thrust chamber assembly was undamaged.

#### Test 1k-2B-128

(C) During the start transient there was a delay of approximately 1/2 sec in the opening of the fuel thrust chamber valve. When the valve did open, an extremely hard start occurred. The O-ring seals between the chamber sections were extruded out of their glands. The injector face was dished inward and the throat insert was broken into four pieces. The fuel line burst just downstream of the venturi used for controlling the fuel flow rate releasing Beryllizine into the test bay. The electrical cables for control and instrumentation were ignited and ultimately destroyed. Because the cables were located adjacent to the hydrogen peroxide feed line, it burst due to the heat and hydrogen peroxide was released into the test bay. After the hard start and concomitant damage and fire, the electrical cabling and the two burst lines were replaced and the test hardware was repaired in preparation for the next test.

#### Test 1k-2B-129

(C) This test used, for the first time, electrical safety switches mounted on the thrust chamber valves. These safety devices were installed to

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

eliminate the danger of delays in valve actuations which occurred during the last test. Each valve had a maximum period in which to open fully or the test would be terminated. The repaired injector and thrust chamber were used for this test. After fire-switch one, the oxidizer valve exceeded its allowable opening time and the test was terminated automatically; however, enough oxidizer and fuel reached the chamber to cause ignition and a moderately hard start. The only damage was a dished central portion of the injector face caused by the hard start and failure of a faulty weld between the hot-core fuel element and the injector housing. The fuel element was designed to support the central portion of the injector face should an overpressure occur in the combustion chamber. Post-test examination revealed little or no penetration of the weld into the housing, providing no support for the face. White beryllium oxide was found inside the thrust chamber and vacuum enclosure, indicating ignition of the beryllium powder prior to shutdown.

#### Test 1k-2B-130

(C) The Centrojet injector equipped with a new porous face and a refurbished thrust chamber were used for this test. The hot-core section of the injector was valved separately from the main manifolds with a mechanically linked bipropellant valve. The objective of the test was to investigate the ignition of only the hot-core section of the injector. The main fuel valve was removed from the system and the lines were capped, and the electrical cable to the main oxidizer valve was disconnected. Almost immediately after fire-switch one the prepurges of gaseous nitrogen started flowing and in approximately 1/4 sec there was an explosion in the combustion chamber. The combustion chamber section next to the injector split longitudinally and was flattened. The combustion chamber and nozzle traveled approximately 150 ft into the vacuum catch tank. The test stand was sheared from its base and irreparably damaged. One closure plate was blown from the vacuum enclosure which surrounded the test stand, and the inrush of air carried the beryllium and beryllium oxide into the catch tank. The valve connecting the exhaust duct to the catch tank was closed immediately, entrapping the contaminants.

(C) A review of the data and hardware after the test indicated that the main oxidizer valve had leaked  $H_2O_2$  into the thrust chamber during the pretest period. The  $H_2O_2$  present in the combustion chamber had combined with a fuel which formed an explosive mixture. The fuel could have been either from the materials of construction of the thrust chamber or Beryllizine that had leaked from the fuel valve. Shortly after the prepurges were activated, the explosive mixture was initiated. All components located within the vacuum enclosure were demolished and had to be replaced. There was no damage outside the enclosure. Subsequent to this explosion, the test program was completely replanned.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(C) An approach was developed to eliminate a recurrence of the explosion experienced during Test 1k-2B-130. An external ignition source, consisting of Alclo igniters, for the hot-core was adopted so that hypergolic ignition need not be relied upon. The igniters would also eliminate hard starts by initiating burning of either the oxidizer or fuel should one reach the chamber first. Dual igniters fired from separate electrical circuits were used to improve the reliability of the ignition system. As an additional precaution, an ignition sensor in the form of an ionization probe was programmed into the start sequence to guarantee proper functioning of the Alclo igniters before the thrust chamber valves could receive their opening signal. New thrust chamber valves were installed along the burst discs located at the injector inlets. Propellant drain valves were located between each thrust chamber valve and its corresponding burst disc. Before each test the drain valves remained open and the pretest purges were used to sweep any leakage of propellant from the outlets of the thrust chamber valves. The drain valves were simultaneously closed when the thrust chamber valves were cycled to the open position. The new drain and thrust chamber valves were mechanically-linked bipropellant units actuated by an air cylinder common to each pair of valves. Two pairs of bipropellant valves were used in the propellant feed circuit to the hot-core section of the Centrojete injector and two were used in the circuit to the main section. The burst discs located at the injector inlets were included for additional assurance that any pretest propellant leakage from the thrust chamber valves would not reach the combustion chamber prematurely and cause a hard start.

(C) When the damage sustained during Test 1k-2B-130 had been repaired, the test facility was re-activated in preparation for checkout testing. The fuel feed system was flowed with Beryllizine to verify proper operation of all components and for calibration of the venturies used to control the rates of fuel flow. The results showed the system was ready for checkout testing.

(C) The oxidizer feed system was filled with 98% hydrogen peroxide and flowed at the design rate of flow and pressure. The system was adequately safe, operating properly and ready for further testing.

(C) All injector components, associated fittings and lines and other miscellaneous items which cannot be exposed to the oxidizer were placed in 70% nitric acid for several days, cleansed with deionized water and immersed in 35% hydrogen peroxide until thorough passivation was assured. This was the same procedure used in preparation for the previous tests. When this task was complete, preparation for checkout testing was begun.

(C) Subsequent to passivation of components and flow testing of the feed systems, the Centrojete injector was assembled and mounted on the test stand. The final plumbing and instrumentation tasks were performed and a new assembly, consisting of thrust chamber and nozzle extension, was installed.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

Tests of the electrical sequencer were performed as a final check on electrical wiring of the patchboard. Opening and closing times and repeatability of valve relays were evaluated and prepurge tests with nitrogen gas were accomplished in order to determine the proper pressure setting.

(C) Three tests using Alclo igniters and the pre- and post-test purges, controlled by the electrical sequencer, were required to achieve acceptable start and shutdown characteristics. The first test was terminated automatically when the ignition sensor (ionization probe) failed to detect ignition and interrupted the firing sequence. Post-test reviews revealed the probable cause was insufficiently ionized nitrogen gas, present in the combustion chamber from the pretest purge, that subsequently prevented the ionization probe from operating properly. A safety shutdown was initiated by the safety timer located within the electrical sequencer.

(C) The ionization probe was removed and burn links were positioned centrally in the exhaust stream of each solid propellant igniter. The links were constructed of lead wire of approximately 0.10-in.-dia, and were wired in series so failure of just one igniter would not interrupt the firing sequence. The second test, using burn links, was successful, but the ignition sensor did not detect ignition until 85 msec after initiation of the Alclo igniters. The time delay was considered excessive so an additional test was conducted using 0.04-in.-dia copper wires as frangible links in place of the burn wires. It was assumed that the copper wires of thinner diameter would break more quickly than the time required to melt the lead wires. A third test was conducted successfully and the reaction time of the ignition sensor was shortened to 10 msec, which was considered acceptable.

(C) The reaction time of the ignition sensor, sequences for start and shutdown and the run duration were considered acceptable and within the conditions established for testing of the hot-core section of the injector.

#### Test 1k-2B-131

(C) This test was to have been a short-duration, checkout type firing using the hot-core section of the Centrojett injector alone for a nominal 400 msec of full chamber pressure. There were two main test objectives, the first to evaluate propellant ignition with external igniters and secondly to investigate combustion of the hot-core alone.

(C) At fireswitch, the Alclo igniters were initiated successfully but the ignition sensor failed to detect ignition and interrupted the firing sequence. Examination of the hardware revealed igniter residue had been deposited on the wall of the combustion chamber during the three igniter tests previously conducted. The deposits provided an electrical leak path between

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

the mounts for the break-wire ignition sensor rendering the sensor inoperative. The shorted condition of the break wires provided a continuous shutdown signal to the electrical sequencer. (Short circuits of this type were detected prior to the remaining tests by measuring the resistance from a break wire to the wall of the combustion chamber at the time of installation.) The short circuit was repaired and preparations were begun for a repeat firing.

(C) At fireswitch, the Alclo igniters were initiated and the ignition sensor detected ignition 10 msec later, permitting the firing sequence to proceed. The thrust chamber valves which control propellant flow to the hot-core were fully open 200 msec after fireswitch and a start spike of 528 psia occurred 475 msec from fireswitch. The chamber pressure was 142 psia near the end of the test and the duration was 350 msec from the time pressure was first measured in the chamber until shutdown occurred.

(C) Post-test examination revealed undamaged hardware and deposits of beryllium oxide inside and outside the thrust chamber.

(C) When the possible cause of the spike in chamber pressure had been evaluated, plans were formulated for the first ignition test with the complete thrust chamber assembly. The spike was not considered a potential hazard or would it have an effect during an ignition test because it was not of sufficient magnitude or duration (10 msec) to cause hardware damage; additionally, it would subside 200 msec prior to entry of the main streams of propellant into the combustion chamber which was considered a safe margin.

#### Test 1k-2B-132

(C) The first ignition test was successfully conducted using the modified Centrojet injector with the 90° mixing and deflecting ring and 40:1 nozzle. Nominal values of chamber pressure and mixture ratio were 1000 psia and 1.2 respectively. A standard length combustion chamber (12.5 in.) was used for this test.

(C) At fireswitch, the Alclo igniters were initiated, the igniter sensor detected an ignition in 10 msec and propellants started flowing to the hot-core 150 msec from the fireswitch. Chamber pressure caused by ignition and combustion of the hot-core occurred 405 msec from fireswitch. The oscillogram showed the ignition and an increase in chamber pressure to 135 psia, where it remained steady for 50 msec. The propellant streams from the main section of the injector entered the combustion chamber 200 msec from ignition of the hot-core. The chamber pressure increased to approximately 950 psia in 150 msec. The duration of steady-state combustion was 1.1 sec.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(C) When the access plate of the enclosure surrounding the test stand was removed, a cloud of beryllium oxide was observed throughout the enclosure and deposits of the oxide coated the interior surfaces and other components in greater quantity than observed subsequent to firings with  $N_2O_4$ /Beryllizine-21. Deposits of beryllium oxide were also found within the thrust chamber during disassembly of the hardware.

(C) Examination of the hardware after the test showed little damage to the nozzle components and increasing evidence of heating of the wall of the combustion chamber up to the injector face. The inner surface of the chamber liner nearest the injector face was unevenly eroded and considerably more damaged than the other liners. The mixing and deflecting ring was burned, with evidence of combustion on the inner, deflecting surface. After cleaning and decontamination, twenty-one pairs of burned spots and one axial gouge, which were spaced quite uniformly, were found on the inner surface. The flat surface of the ring, nearest the combustion chamber, showed evidence of the same heating pattern in a corresponding position. The central axes of the burned spots were near the oxidizer orifices located in the main section of the injector. The porous face of the hot-core section of the injector was missing and the surfaces behind and adjacent to the face were badly burned. Further examination revealed the injector in repairable condition.

(C) The thrust chamber was in satisfactory condition for an additional test with the exception of the section of combustion chamber nearest the injector. It was refurbished with a new liner of ablative material in preparation for the next firing.

(C) This test resulted in the highest specific impulse known delivered by earth-storable propellants (see Table XXXIX and Figures 45 and 46).

(C) Although the previous test was successful from the ignition and combustion point of view, burning of the injector face required modifications to the hardware and test conditions to avoid such damage during the remaining tests. Detailed analyses of the injector damage and flow tests with water indicated that the damage may have been caused, at least in part, by leakage of the hydrogen peroxide around the porous face. Monopropellant decomposition of the peroxide in the porous material may have contributed to its failure. The test conditions were examined closely and modifications were planned which would further enhance durability of the injector face.

(C) During a routine inspection of the equipment after this test, fatigue cracks were observed on the outer wall of the oxidizer tank. The tank was replaced with a tank of similar size which had previously been in peroxide and  $N_2O_4$  service. The tank was filled with 70%  $HNO_3$  and allowed to stand for one hour. The tank was drained, rinsed with deionized water and filled with 98%  $H_2O_2$ . The vent valve was blocked open to permit escape of any evolved gas. The tank was inspected at approximately 1/2-hr intervals for the next eight hours for changes in temperature or signs of gas evolution.



## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) Approximately 5 $\frac{1}{2}$  hr after being filled with peroxide, the oxidizer feed system was severely damaged when the tank burst due to explosive decomposition of the peroxide. Damage was confined to the oxidizer feed system and its supporting structure; a storage cabinet containing valves, tools and other expendable components; and to the walls and roof of the test bay. The damage was repaired and the test equipment reassembled for resumption of testing.

(C) An Aerojet committee conducted an investigation of the explosion, but was unable to establish a specific cause. However, two factors which may have had an important bearing on the cause were that (1) due to poor welding technique there was a crevice in one of the welded fittings attached to the bottom of the tank that may have escaped thorough cleaning and passivation and (2) one of the passivation steps was omitted from the preparation of the peroxide tank for service. The step involving treatment with stabilized 35% hydrogen peroxide was omitted because the tank had previously been in peroxide service and further passivation with the 35% solution was believed not to be necessary.

#### Test 1k-2B-133

(C) This test was the first conducted using the rebuilt oxidizer feed system which was damaged in the oxidizer tank explosion. Nominal conditions for the test were 1000 psia chamber pressure, 1000 pounds of thrust and a mixture ratio of 0.6 to 0.7. The Centrojet injector was used with a face of very porous material, no oxidizer orifices for the hot-core and a propellant convergence angle of 90 degrees. The overall test duration was to be approximately one second.

(C) Post-test inspection of the hardware revealed heat-caused discoloration of the porous face around its periphery, and mild erosion on the deflector ring in a scalloped pattern. The internal surfaces of the thrust chamber were uneroded and evenly covered with beryllium oxide. The throat was circular and had increased in diameter by 0.006 in. Caked deposits of beryllium oxide were observed on the divergent surface of the nozzle.

(C) Review of the oscillogram revealed low frequency combustion instability. The frequency was approximately 120 cps and the amplitude was  $\pm$  300 psia. The instability was caused by acoustic coupling of the combustion process with the long oxidizer feed line which delivers oxidizer from the thrust chamber valve to the hot-core/porous-face section of the injector. The pressure drop across the highly porous face used for this test was too low (47 psi or 5% of chamber pressure) and was the primary cause for the instability.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### Test 1k-2B-134

(C) The conditions for this test were the same as for Test 1k-2B-133. The goals were to eliminate the low frequency combustion instability and to increase the delivered specific impulse at low mixture ratio. The porous face for the Centrojet injector was replaced with an insert fabricated from low porosity material and containing the drilled orifices for the oxidizer portion of the hot-core. It was postulated that the higher pressure drop of the porous face would prevent the combustion instability, and the use of the original hot-core configuration would improve combustion and increase specific impulse. The thrust chamber was fitted with a new throat insert in preparation for this test.

(C) After the test the oscillogram revealed smooth combustion with no trace of low frequency instability and the final data revealed a .9% improvement in performance. When the hardware was removed from the stand, the porous face was found to be slightly discolored by heat and mildly eroded around the central swirler. The throat diameter had decreased by .002 in. and beryllium oxide was found inside the chamber and caked on the surface of the divergent section of the nozzle.

#### Test 1k-2B-135

(C) This test was conducted with a long combustion chamber at the same conditions of the previous two tests. The Centrojet was fitted with a new face of low porosity, including the hot-core oxidizer orifices. The thrust chamber was assembled on the stand using nine chamber sections and the nozzle from the preceding test. The length of the chamber was 22.5 in., measured from the injector face to the nozzle entrance, whereas the corresponding distance used for previous tests was 12.5 in. The purpose of this test was to determine if performance could be improved by increasing the chamber length.

(C) The data resulting from the test revealed the performance was 3.1% higher than that measured during the previous firing, and the oscillogram showed smooth combustion, with no trace of low frequency instability. The porous face was less discolored than observed after the previous two tests, and there was a small spot of erosion on the porous face adjacent to an oxidizer orifice. The throat diameter decreased by .002 in. Deposits of beryllium oxide were found in the chamber and on the nozzle surfaces.

#### Test 1k-2B-136

(C) The purpose of this test was to measure the performance at a mixture ratio of 0.9, which is intermediate between those used in the previous four tests. Test 132 was conducted at a mixture ratio of 1.1 and performance

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

efficiency was 90.3%. The efficiency ranged from 83 to 87% in Tests 133 through 135, in which the ratios averaged 0.7. The theoretical combustion temperature at a mixture ratio of 0.7 is 6010°R, whereas at a ratio of 1.1, it is 6150°R. On the other hand, the concentration of water vapor in the combustion products increases from 8 mole percent at 0.7 to 24 mole percent at 1.1. Because the change in water concentration in the combustion zone is more significant than the change in temperature, it is believed that combustion efficiency improves with increased water concentration rather than increased temperature. Additional evidence has been recently reported which supports the theory of the role of water vapor in the ignition and combustion of beryllium-containing fuels (Reference 20). In view of this, a mixture ratio of 0.9 was selected for Test 136 to determine if the relation of performance efficiency to water concentration could be confirmed. Figure 47 presents the relationship of combustion temperature and water concentration with mixture ratio for the  $N_2O_4$ /Beryllizine-28G propellant system.

(C) The Centrojet injector with a new porous face, and a short chamber which had been refurbished with new inserts were mounted on the test stand.

(C) After the test, the oscillogram showed stable combustion and the data revealed a performance efficiency of 89.5%. The porous face was badly eroded around the central swirler and discolored by heat. The other injector and chamber components were in the same condition as after the previous tests.

#### d. Results of Testing

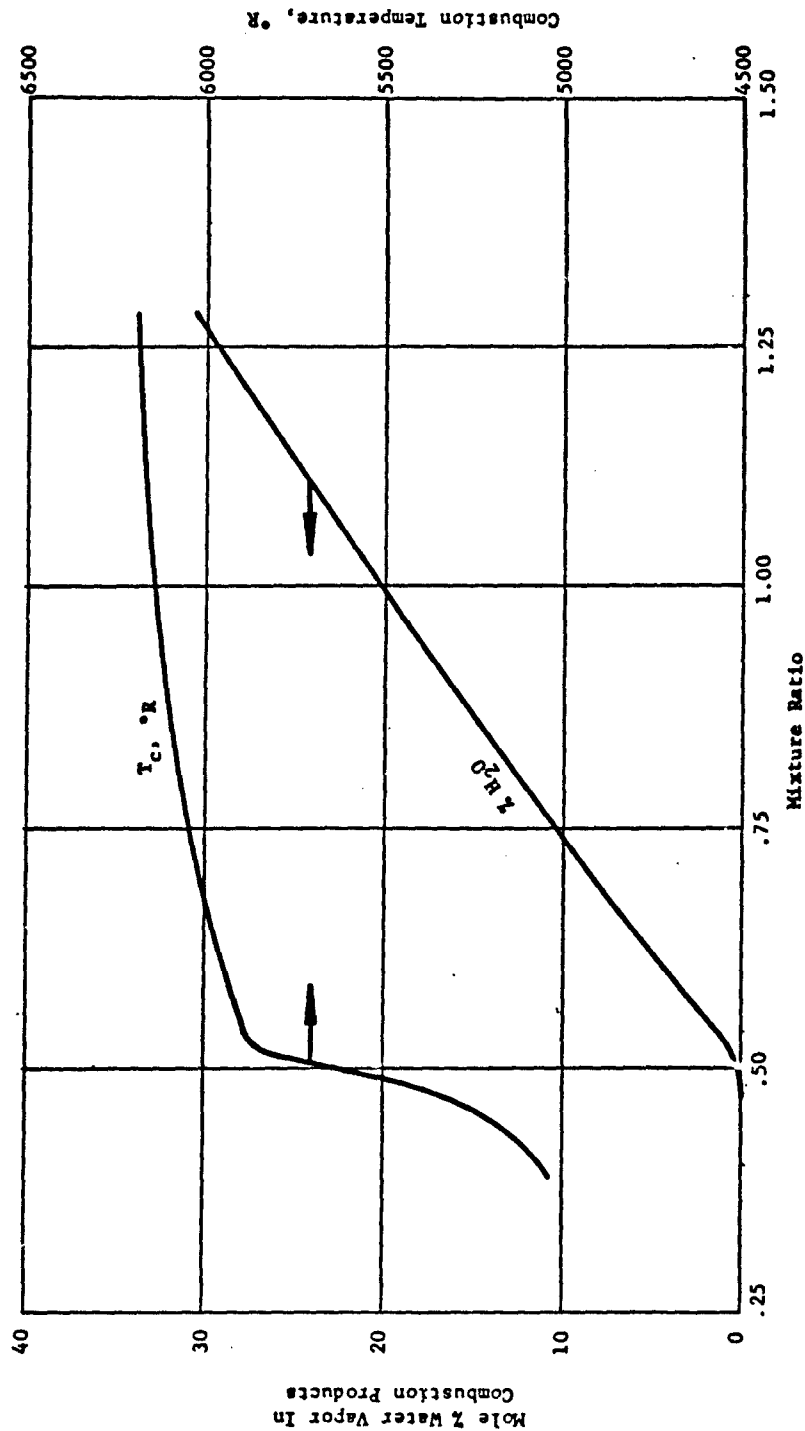
(C) The results of testing with both the  $N_2O_4$ /Beryllizine-28G propellant combinations were processed in the same manner. Particular emphasis was placed on analyses of experimental data, exhaust samples and nozzle losses attributable to solid-particle flow. A discussion of the treatment of experimental results and the methods of data analysis is contained in the following sections.

#### (1) Analysis of Experimental Data

(U) The experimental data consisted of measurements of thrust, chamber pressure, propellant flow rates and temperatures, injector pressures, the pressure within the vacuum enclosure and other pressures throughout the propellant feed systems. These data were recorded on an oscillograph to reveal trends and relationships, and the data for which maximum precision was required were recorded and processed by a millisecondic system. Throat diameters were accurately measured before and after each test and these values were used to compute throat areas and area ratios for analytical purposes.

CONFIDENTIAL

Report AFRPL-TR-66-230



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Figure 47. Relationship of Combustion Temperature and Water Concentration with Mixture Ratio for the 93%  $H_2O_2$ /Beryllizine-28 Propellant System

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## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(U) The experimental values of specific impulse, characteristic velocity and thrust coefficient were calculated by hand preparatory to computer processing of these data. An existing IBM 7094 computer routine was used for data processing. The computer calculated the theoretical conditions corresponding to the exact experimental conditions of each test based upon the chemical compositions and performance parameters generated earlier in the program. The theoretical data were then compared with the corresponding empirical values and percentages of  $I_s$ ,  $c^*$  and  $C_F$  were obtained.

(U) The experimental data from the tests with the  $N_2O_4$ /Beryllizine-21 propellant system may be found in Tables XXXVII and XXXVIII. Graphical relationships are presented in Figures 42, 43 and 44. The data resulting from tests with 98%  $H_2O_2$ /Beryllizine-28 may be found in Table XXXIX and Figures 45 and 46.

#### (2) Analysis of Exhaust Particles

(U) A probe for the purpose of collecting solid-phase exhaust samples was located in the duct connected between the vacuum enclosure surrounding the test stand and the vacuum catch tank. Wipe samples were also taken from an interior surface of the test stand enclosure. The surface was thoroughly cleaned before a test from which exhaust samples were desired.

(U) The samples collected with the probe and obtained from inside the vacuum enclosure were dispersed separately in light mineral oil and observed under a microscope. Observations were made of the size and shape of the particles. When it was determined that the sizes and shapes of particles were not changing from test to test, a particle count was made for each of the three propellant systems tested.

(C) The microscopic observations revealed that the collected particles were similar throughout the test program with Beryllizine. The particles of beryllium oxide observed after the tests with  $N_2O_4$  and 98%  $H_2O_2$  appeared white and irregularly shaped with jagged edges. The particles of aluminum oxide observed after testing with  $N_2O_4$ /Alumizine-43G were also white, but spherically shaped with smooth edges. Particle counts made after the firings with  $N_2O_4$ /Beryllizine-21,  $N_2O_4$ /Alumizine-43G and 98%  $H_2O_2$ /Beryllizine-28 revealed the majority of all observed particles were less than 2 microns in diameter. The percentages of oxide particles from the three systems tested which were less than 2 microns in diameter are as follows:  $N_2O_4$ /Beryllizine-21, 83.3%;  $N_2O_4$ /Alumizine-43G, 90.5%  $H_2O_2$ /Beryllizine-28, approximately 80%. X-ray analyses of the samples obtained from the tests with  $N_2O_4$  and 98%  $H_2O_2$  revealed the deposits to be 94 and 93% beryllium oxide, respectively.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

#### (3) Analysis of Solid-Particle Flow Losses

(U) These losses were calculated with the aid of an axisymmetric, two-phase flow, nozzle evaluation computer program. The losses were found to vary from about 5% for a one-micron particle diameter to about 14% for eight-micron diameters. Following will be found a discussion of the scope of the study, the methods used and a discussion of the results.

(C) The investigation was conducted for the  $N_2O_4$ /Beryllizine-21 propellant system operating at a chamber pressure of 1000 psia over the mixture ratio range of 0.536 to 1.250. The theoretical data for chemical compositions and performance parameters generated earlier in the program were used to compute the losses in theoretical performance as a function of mixture ratio for an expansion ratio of 40:1. The concentration of particles in the exhaust stream varied from 0.37 pounds of particles per pound of exhaust mixture at a mixture ratio of 0.536 to 0.25 lb/lb at a mixture ratio of 1.25. All flow calculations were performed for the particular 40:1 nozzle configuration used during this program.

(U) Various computer programs are available at Aerojet for analysis of the gas flow in rocket nozzles; however, only two consider the effects of particles in the gas stream. The program selected for this study calculates the gas/particle flow field in a given nozzle considering shifting equilibrium of the gas, freezing of the particles, axisymmetric flow and the interrelationship between the particles and the gas. No friction of heat transfer losses are calculated and ideal combustion and mixture-ratio distribution are assumed. Performance values, calculated by the program for various assumed particle sizes are shown in Figure 48 compared with theoretical specific impulse.

(U) Another nozzle analysis program was used to calculate performance for an assumed ideal gas with flow frozen at the chamber conditions. This curve is also presented in Figure 48. The principal reason for these calculations was to obtain geometry losses, which were calculated by comparing axisymmetric frozen-flow performance to that for one-dimensional frozen flow, so they could be subtracted from the axisymmetric particle losses.

(U) The curves show that particles of 1 to 2 microns in diameter, which were observed after the experimental firings, result in losses on the order of 5 to 8%. When evaluating these predictions, it should be kept in mind that particle losses have been shown to be quite dependent on the size of the nozzle with the smaller sizes evidencing substantially greater losses than the larger ones. Considering this fact, the values appear reasonable.

CONFIDENTIAL

Report AFRPL-TR-66-230

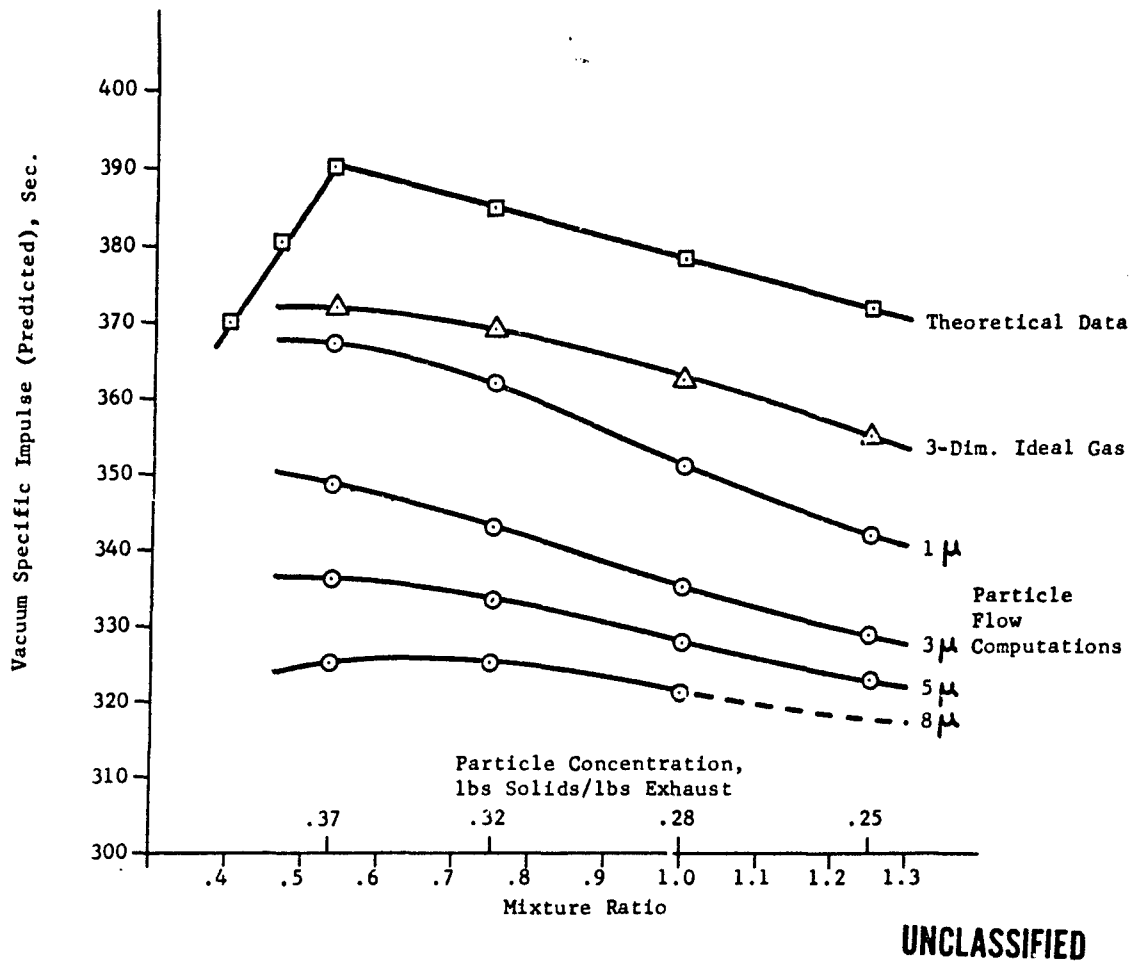


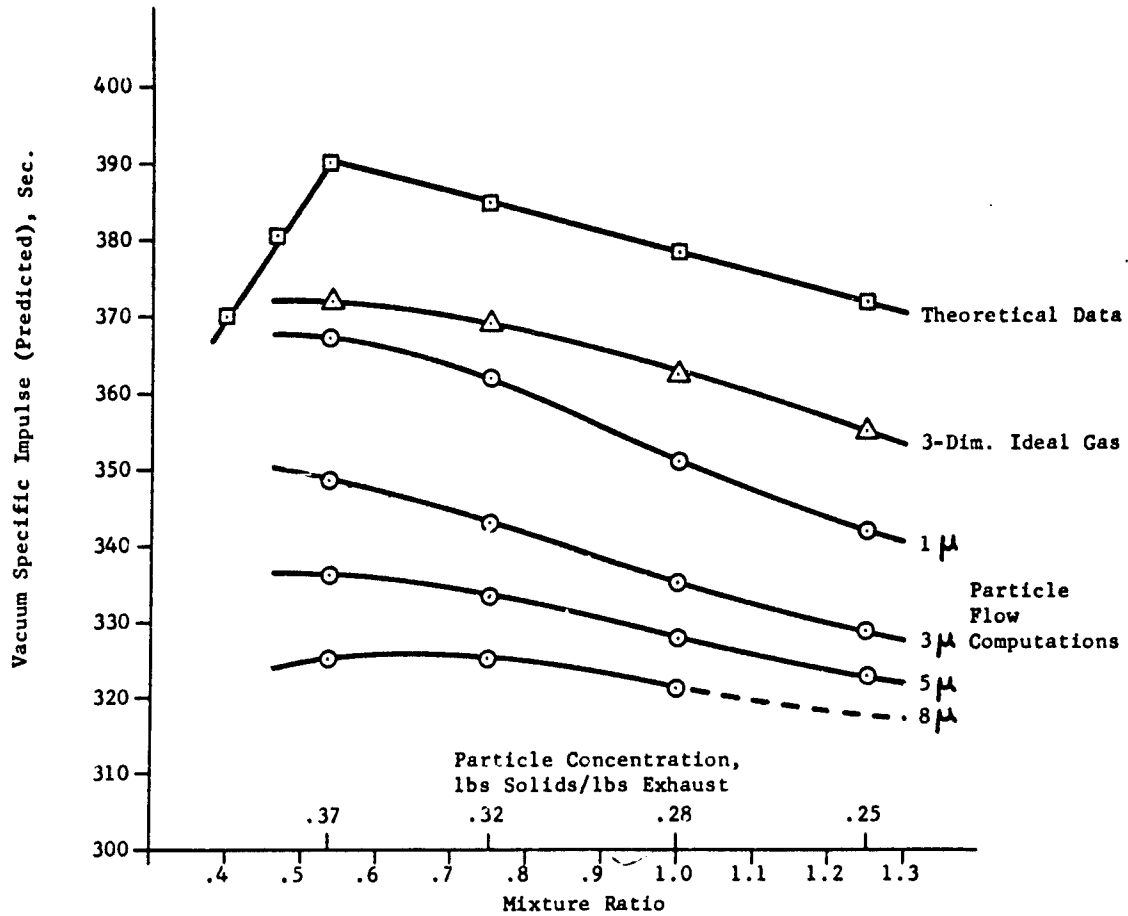
Figure 48. Vacuum Specific Impulse versus Mixture Ratio as Predicted by Thermochemical, Ideal-Gas and Particle Flow Methods

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Report AFRPL-TR-66-230



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Figure 48. Vacuum Specific Impulse versus Mixture Ratio as Predicted by Thermochemical, Ideal-Gas and Particle Flow Methods

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## CONFIDENTIAL

Report AFRPL-TR-66-230

### III, B, Engineering Investigations (cont.)

(C) If the results of testing with the  $N_2O_4$ /Beryllizine-21 propellant system are corrected for losses attributable to solid-particle flow, the achieved efficiency of specific impulse will increase from 86.3% to approximately 92 to 93%.

#### C. PROPELLANT HANDLING AND OPERATING PROCEDURES

(C) An additional objective of this program was to demonstrate that beryllium can be safely handled in laboratory and engineering operations using the procedures described in the letter to Captain Owen Kittelstad, Edwards Air Force Base, from Mr. Herbert Dankman of the Industrial Hygiene Department, Aerojet-General Corporation, dated 16 April 1965.

(C) During this program approximately 750 lbs of Beryllizine propellant were prepared and extensive laboratory investigations using beryllium powder were conducted. The beryllium handling procedures were found to be effective in all operations. In order to benefit from the additional experience gained during this program, the safety and handling procedures were continuously reviewed and improved to eliminate any deficiencies that became apparent. Regular inspections of masks and respirators were initiated to ensure that all personnel were properly fitted, that all masks were in good condition, and that proper filters and canisters were being used. Pressure relief valves were installed on all glove boxes and processing equipment that were operated at reduced pressures. These valves vent through an absolute filter system and prevent contamination of the atmosphere caused by inadvertent pressurization of laboratory equipment containing toxic materials.

(C) Thirty-seven 1-K engine firings were conducted. Two malfunctions occurred in the firing tests which resulted in the release of small quantities of beryllium to the atmosphere. No personnel were exposed to beryllium, and the facilities and equipment were easily decontaminated. This experience confirms that beryllium can be safely handled using the procedures referenced above.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### SECTION IV

#### CONCLUSIONS AND RECOMMENDATIONS

##### A. CONCLUSIONS

###### 1. Chemical Laboratory Investigations

(C) The prototype Beryllizine-21 and -28 prepared for the engineering phases of this program met all of the requirements with regard to composition, density, flow properties and mechanical stability; however, the bulk growth and gas evolution rates were high and it was necessary to limit storage to 2 months.

(C) Investigations of the compatibility of ungelled hydrazine with untreated beryllium powder showed that the purest beryllium powders are the most compatible with hydrazine and that magnesium and beryllium carbide appear to be most responsible for high gas evolution rates.

(C) Treatment of the various beryllium powders with 5% nitric acid, 30% hydrogen peroxide and anhydrous hydrazine were ineffective in reducing the gas evolution rates of ungelled mixtures of beryllium and hydrazine, as were heat-treatments or coatings on the beryllium powders. Of the treatments given the beryllium powders only the water extraction and alcoholic potassium hydroxide treatments were effective. The water treatment of chromium-passivated beryllium with low impurity content was successful in producing a metal powder that, when mixed with hydrazine, met the program goal for gas evolution. The alcoholic potassium hydroxide treatment was not as effective as the water treatment.

(C) Investigation of the gas evolution rates of beryllium-hydrazine mixtures gelled with Carbopol-940 indicated that the Carbopol adversely affects the compatibility of the mixture. Substitution of the Aerojet gelling agents TPAP and TPAP-12 eliminated this problem. Beryllizine fuels prepared with TPAP or TPAP-12 and water-treated chromium-passivated beryllium powder closely approximated the program goal for gas evolution rate.

(C) Experience gained in the preparation of nearly 750 lb of propellant demonstrated that Beryllizine can be reliably and reproducibly prepared using the standard mix procedure developed for Alumizine propellant.

###### 2. Engineering Investigations

(C) The comparison of data obtained with Beryllizine-21 and Alumizine-43G using the ASTM D-1092 capillary viscometer showed that the flow characteristics of the two fluids are similar in the laminar regime. The

## CONFIDENTIAL

Report AFRPL-TR-66-230

### IV, A, Conclusions (cont.)

capillary-viscometer data and the flow-loop data obtained with Beryllizine-21 were consistent. The similarity of data for Alumizine-43G and Beryllizine-21 indicates that the flow characteristics of the fluids are the same in the turbulent regime. The transitional points of both fluids were found to occur at nearly identical shearing rates, which further indicates similarity in flow characteristics.

(C) The hydraulic characteristics of Beryllizine-21 and Beryllizine-28 chosen for engineering design purposes were based upon previous experience with Alumizine-43G. Since the assumptions were correct, it is possible to predict flow characteristics with enough precision for the proper hydraulic design of injectors.

(C) Because problems of erosion, clogging, or fuel separation were not encountered, conventional techniques can be used for the design of flow passages for the prototype Beryllizine-21 and -28; however, provisions must be made for cleaning residual fuel from the internal cavities.

(C) The hot-core designs of both the modified Centrojet and the second injector produced conditions which ignited the beryllium constituent of Beryllizine-21, but the Centrojet gave the best performance efficiency.

(C) Substitution of the smaller diameter FP-555 beryllium powder for the FP-1755 powder in the Beryllizine-21 had no noticeable effect on either the ignition characteristics or the performance efficiency obtainable from this propellant system.

(C) Since the performance efficiency demonstrated with  $N_2O_4$ /Beryllizine-21 (86.3% of theoretical specific impulse, uncorrected for two-phase flow) is nearly identical to that obtained with  $N_2O_4$ /Alumizine-43G (85.3%) at the same conditions, the 1000-lb thrust chamber assembly was operating with acceptable efficiency. On the basis of this comparison, the high percentages of theoretical performance previously achieved with  $N_2O_4$ /Alumizine-43G at higher thrust levels can be anticipated for the  $N_2O_4$ /Beryllizine-21 system.

(C) The hot-core technique for ignition of beryllium was demonstrated with the  $H_2O_2$ /Beryllizine-28 system. Beryllizine oxide was found in the combustor chamber after the tests in which the Alclo igniters were not used.

(C) The test results show that the percentage of theoretical performance for the  $H_2O_2$ /Beryllizine-28 system rises with increasing mixture ratio. It is postulated, on the basis of existing experimental data, that this effect results from increased water concentration in the combustion products rather than increased combustion temperature.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### IV, A, Conclusions (cont.)

(C) The test conducted with the 22.5-in.-long combustion chamber showed an improvement in performance efficiency of  $H_2O_2$ /Beryllizine-28 at essentially the same mixture ratio, when compared with tests conducted with the 12.5-in.-long chamber.

#### 3. Propellant Handling and Operating Procedures

(C) The beryllium handling procedures used in this program were adequate for protecting all personnel as well as for preventing contamination of air, water, or soil.

### B. RECOMMENDATIONS

#### 1. Chemical Laboratory Investigations

(U) In order to provide a theoretical basis for the information developed under this program a fundamental investigation of the gas evolving reactions in Beryllizine should be conducted. The information from this fundamental investigation, along with that from the program described in this report, would be used to (1) elucidate the nature of the decomposition reaction of hydrazine and the effects of the other fuel components and impurities on it, and (2) define the reactions that occur between the various propellant components and impurities.

(C) The results of this fundamental investigation should then be used to improve the Beryllizine developed during this program. This would be accomplished by (1) optimizing the water-treatment of beryllium powders or developing an alternate treatment, (2) purification of the components to remove or passivate the impurities which cause incompatibility and (3) development of formulations based on the TPAP or TPAP-12 gelling agents or modifications of these gelling agents.

(U) Based on these investigations, development of improved Beryllizine fuels that meet the requirements for an advanced propulsion system should be completed by optimizing the flow properties and completing characterization of the fuels, including long-term storability.

#### 2. Engineering Investigations

(U) Future flow loop testing should be conducted in temperature-controlled equipment. It is extremely difficult to correlate data from a series of flow tests if the propellant properties do not remain constant.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### IV, B, Recommendations (cont.)

(C) Based upon the results of this program, it is recommended that the flow properties of Alumizine-43G be used to predict the behavior of Beryllizine-21 in both the laminar and turbulent regions of flow as long as the yield stress values of both fluids are similar.

(C) It is recommended that an investigation be conducted to determine the effect of water vapor on the ignition and combustion of beryllium-containing fuels. The results of this program provide evidence that the conditions for beryllium-particle ignition become less stringent, and combustion becomes more efficient with increased water vapor in the combustion zone.

(C) The high specific impulse values achieved with the  $N_2O_4$ /Beryllizine-21 and 98%  $H_2O_2$ /Beryllizine-28 propellant systems demonstrate the feasibility of these gelled-metalized propellant combinations. It is recommended that these propellant systems be investigated at the 5000-lb thrust level, with particular emphasis on thrust chamber design. Design criteria to improve injector durability should be developed along with analytical and empirical studies of combustion chamber and nozzle geometries.

## CONFIDENTIAL

Report AFRPL-TR-66-230

### SECTION V

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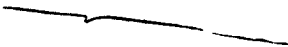
Report AFRPL-TR-66-230

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UNCLASSIFIED

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**CONFIDENTIAL**

Report AFRPL-TR-66-230

APPENDIX A

BERYLLIZINE FUEL CRITERIA (u)

Page 165

**CONFIDENTIAL**

(This page is Unclassified)

**CONFIDENTIAL**

Report AFRPL-TR-66-230, Appendix A

**CONFIDENTIAL**

(This page is Unclassified)

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix A

## TABLE OF CONTENTS

	<u>Page</u>
1.0 <u>SCOPE</u>	168
1.1 Purpose	168
2.0 <u>APPLICABLE DOCUMENTS</u>	168
2.1 Military Specifications	168
2.2 Contractor Specifications and Standards	169
2.3 Other Documents	169
3.0 <u>CRITERIA AND REQUIREMENTS</u>	170
3.1 Propellant Characteristics	170
3.1.1 Mechanical Stability	170
3.1.2 Chemical Stability	170
3.1.3 Maximum Yield Stress	170
3.1.4 Shear-Thinning Rheology	170
3.2 Propellant Composition	172
3.2.1 Component Concentrations	172
3.2.2 Components Property Restrictions	172
3.3 Essential Physical Properties	173
3.3.1 Density	173
3.3.2 Yield Stress	173
3.3.3 Stress-Strain Ratio	174
3.3.4 Smoothness	174
3.4 General Properties	174
4.0 <u>REQUIRED TEST EQUIPMENT AND PROCEDURES</u>	175
4.1 Equipment	175
4.2 Standard Test Conditions	175
4.3 Standard Test Procedures	175
5.0 <u>QUALITY ASSURANCE PROVISIONS</u>	176
6.0 <u>PREPARATION FOR SHIPMENT</u>	176
7.0 <u>NOTES</u>	177
7.1 Intended Use	177
7.2 Safety Precautions	177

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix A

### 1.0 SCOPE

#### 1.1 Purpose

(C) The purpose of this document is to specify the propellant criteria and limits required to define the two gelled beryllium-hydrazine fuels designated Beryllizine-21 and Beryllizine-28. These criteria provide the bases for the development of the fuels and an outline of the methods used in characterizing them. The criteria may be used to compare Beryllizine with other propellants and to relate Beryllizine with propulsion systems. These criteria and corresponding limits will be revised as necessary.

#### 1.2 Summary of Fuel Definition Requirements

(U) The items that must be determined, specified, and/or controlled in order to adequately define the Beryllizine fuels are as follows:

##### 1.2.1 Fuel Criteria

Propellant Characteristics

Propellant Composition

Essential Physical Properties

##### 1.2.2 Test Equipment and Procedures

Required Equipment

Standard Test Conditions

Standard Test Procedures

##### 1.2.3 Quality Assurance Provisions

### 2.0 APPLICABLE DOCUMENTS

(U) The following documents shall form a part of this document to the extent specified herein.

#### 2.1 Military Specifications

MIL-P-26556 Propellant, Hydrazine

MIL-P-27412 Propellant, Alumizine-43G

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix A

(U) 2.2 Contractor Specifications and Standards

Aerojet-General Corporation

Specifications

AGC-44184	Propellant, Alumizine-43G
AGC-44185	Carbopol-940 Power
AGC-44186	Aluminum Powder (soluble-electrolytes test, para. 4.7.3)

Standards

AGC-STD-4874	Determination of Dielectric Constant of Hydrazine
AGC-STD-4878	Determination of Yield Stress of Alumizine-43G using the Rising Sphere Rheometer
AGC-STD-4876	Determination of Density of Alumizine-43G
AGC-STD-4877	Determination of Brookfield Viscometer Dial Readings for Alumizine-43G
AGC-STD-4875	Determination of Carbopol-940 in Alumizine-43G
AGC-STD-4873	Determination of Hydrazine and Water in Alumizine-43G

(U) 2.3 Other Documents

Method of Testing Viscosity, ASTM D-1092

Aerojet-General Laboratory Procedure for Measuring High-Shear  
Viscosity with the Ferranti-Shirley Viscometer

MIL-Standard-129                      Marking for Shipment and Storage

I.C.C. 49CFR 71-78                      I.C.C. Regulations for Transportation  
of Explosives and Dangerous Articles

I.C.C.-5C

I.C.C.-193-W

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix A

### 3.0 CRITERIA AND REQUIREMENTS

#### 3.1 Propellant Characteristics

(C) The objective of the preparation of Beryllizine is to obtain a stable mixture of beryllium powder in gelled hydrazine, which fuel shall exhibit required characteristics. These characteristics are essential criteria for the application of Beryllizine in propulsion and missile systems. The Beryllizine must meet the following criteria and limits:

##### 3.1.1 Mechanical Stability

(C) Static Limits - The fuel shall be mechanically stable throughout the temperature range from 35° to 120°F, and there shall be no liquid separation or settling of beryllium during the storage period of one year at ambient temperatures.

(U) Dynamic Limits - The fuel shall be mechanically stable during a one-year storage period when subjected at quarterly intervals to vibrations of \_\_\_\_ g over a frequency range from \_\_\_\_ to \_\_\_\_ cps for \_\_\_\_ hours, and to a longitudinal acceleration of \_\_\_\_ g for \_\_\_\_ minutes.

##### 3.1.2 Chemical Stability

(C) The fuel shall be chemically stable and there shall be no sudden pressure increase in storage vessels during long-term storage.

(C) The maximum limit on the gas evolution rate shall be  $1.5 \times 10^{-4}$  cc(nTP)/lb-min for fuel under storage at 77°F.

(C) The maximum limit on the bulk growth rate shall be  $5 \times 10^{-6}$  %/min for fuel under storage at 77°F.

##### 3.1.3 Maximum Yield Stress

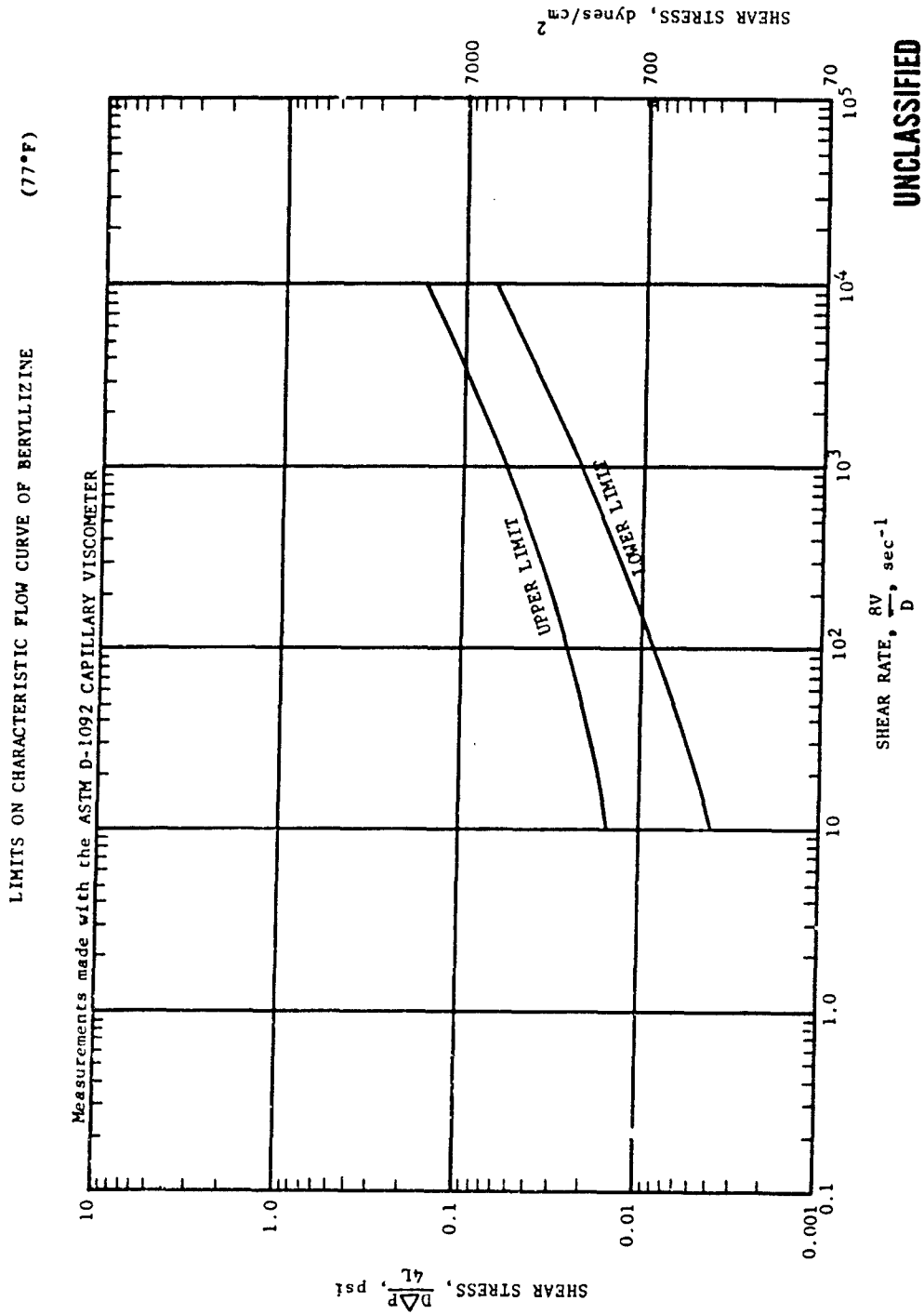
(C) The yield stress of fuel shall not exceed 2000 dynes/cm<sup>2</sup> at 77°F.

##### 3.1.4 Shear-Thinning Rheology

(U) The fuel shall be a shear-thinning fluid. As measured with the ASTM-D-1092 capillary viscometer, the fuel shall have a characteristic flow curve within the shear rate region from 10 to 10,000 sec<sup>-1</sup> that falls within the limits specified on the attached standard figure, Figure 1.

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Report AFRPL-TR-66-230, Appendix A



CONFIDENTIAL

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Report AFRPL-TR-66-230, Appendix A

(U) The fuel shall exhibit a Brookfield limiting-flow characteristic within the limits described on the Standard Figure incorporated in the propellant specification, MIL-P-27412 (USAF).

### 3.2 Propellant Composition

#### 3.2.1 Composition Concentrations

(C) The major components of Beryllizine are beryllium powder, hydrazine and the gelling agent, Carbopol-940. The water impurity shall be considered in the composition. The composition of the two Beryllizine fuels shall be as follows:

A.	<u>BERYLLIZINE-21</u>		
		<u>CONCENTRATION, percent</u>	<u>TOLERANCE, limit</u>
	Hydrazine	77.5 minimum	
	Beryllium	21.0	0.5
	Carbopol-940	0.55 to 0.60	
B.	<u>BERYLLIZINE-28</u>		
		<u>CONCENTRATION, percent</u>	<u>TOLERANCE, limit</u>
	Hydrazine	70.5 minimum	
	Beryllium	28.0	0.5
	Carbopol-940	0.50 to 0.60	
	Water	0.8 maximum	

#### 3.2.2 Component Property Restrictions

(U) The properties of the components for Beryllizine shall be limited by the restrictions specified below. These restrictions on the components are required to ensure that there shall be no deleterious effects on the quality of Beryllizine.



## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix A

(U) 3.2.2.1 Beryllium Powder

(To be determined)

(U) 3.2.2.2 Hydrazine

(U) The hydrazine used in the propellant shall be in accordance with specifications MIL-P-26536 and MIL-P-27412 (USAF), which include the qualities listed below:

(a) The water concentration shall not exceed 1.0 percent.

(b) The hydrazine assay shall be a minimum of 98.5 percent.

(c) A dielectric constant limit of 47 maximum shall be required of the hydrazine as tested in accordance with MIL-P-27412 (USAF).

### 3.3 Essential Physical Properties

(U) These properties are essential to the complete characterization of a propellant, and they are important as propellant criteria for specifying the Beryllizine fuels. The properties shall be in accordance with the following requirements for the fuels:

#### 3.3.1 Density (77°F)

(C) The density as measured with the specified procedure in Paragraph 4.0 shall be:

	<u>BERYLLIZINE-21</u>	<u>BERYLLIZINE-28</u>
a. gm/cc.	1.105 to 1.120	1.145 to 1.160
b. lb/ft <sup>3</sup> ,	68.98 to 69.92	71.48 to 72.42

#### 3.3.2 Yield Stress (77°F)

(C) The yield stress as measured in accordance with the procedure specified in Paragraph 4.0 shall be:

	<u>BERYLLIZINE-21</u>	<u>BERYLLIZINE-28</u>
dynes/cm <sup>2</sup> ,	1400 to 2000	1400 to 2000

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Report AFRPL-TR-66-230, Appendix A

BERYLLIZINE-21 BERYLLIZINE-28

Specific conductivity

Compressibility

Evaporation rate

Impact Stability  
(Drop Weight)

Explosive Sensitivity  
(Card-gap)

#### 4.0 REQUIRED TEST EQUIPMENT AND PROCEDURES

##### 4.1 Equipment

(U) The following equipment shall be used in performing the acceptance tests for the Beryllizine fuels:

a. Rising Sphere Rheometer. The instrument shall be as described in MIL-P-27412 (USAF) for Alumizine propellant.

b. Ferranti-Shirley Viscometer. This instrument shall be as manufactured by the Ferranti-Electric Company, Plainview, New York.

c. Brookfield Viscometer. The instrument shall be as described in MIL-P-27412 (USAF) for Alumizine propellant.

d. ASTM D-1092 Capillary Viscometer. This viscometer shall be as specified in test method ASTM D-1092.

e. High-Frequency Oscillometer. This instrument shall be the unit, Sargent Model 5, as described in MIL-P-27412 (USAF).

##### 4.2 Standard Test Conditions

(U) The standard test conditions shall be 77°F and one-atmosphere pressure (14.7 psia). A protective cover of nitrogen gas shall be used above all propellant to avoid exposure to air. In performing tests, it is essential to minimize exposure of samples to air because the fuel is affected by carbon dioxide and moisture.

##### 4.3 Standard Test Procedures

(U) The standard test procedures listed below will be used in conducting tests on the Beryllizine fuels.

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Report AFRPL-TR-66-230, Appendix A

### 3.3.3 Stress-Strain Ratio (77°F)

(C) The stress-strain ratio shall be measured at a shear ratio of  $10,000 \text{ sec}^{-1}$  in accordance with the procedures specified in Paragraph 4.0, and shall be as follows:

	<u>BERYLLIZINE-21</u>	<u>BERYLLIZINE-28</u>
a. Ferranti-Shirley Viscometer	45 to 70 cp	___ to ___ cp
b. ASTM D-1092 Viscometer	55 to 85 cp	___ cp

### 3.3.4 Smoothness

(U) The quality of Beryllizine-21 and of Beryllizine-28 shall be smooth as measured in accordance with the specified procedure in "Tentative Military Specification, Propellant, Beryllizine."

### 3.4 General Properties

(U) The following are general physical properties of the fuels which are useful information for design and handling purposes, and as such, they are not specification properties. The properties are listed below for information only.

#### BERYLLIZINE-21   BERYLLIZINE-28

Freezing point, °F

Vapor pressure,  
(77°F) mm Hg

psi

Flash Point, °F

Fire Point, °F

Coefficient of

Thermal Expansion, °F<sup>-1</sup>

Dielectric Constant

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix A

- 4.3.1 Density - MIL-P-27412 (USAF)
- 4.3.2 Yield Stress - MIL-P-27412 (USAF)
- 4.3.3 Gas Evolution Rate
- 4.3.4 Bulk Growth Rate
- 4.3.5 Stress-Strain Ratio
  - a. Ferranti-Shirley Viscometer Procedure
  - b. ASTM D-1092 Viscometer - ASTM D-1092
- 4.3.6 Brookfield Limiting-Flow Characteristic - MIL-P-27412 (USAF)
- 4.3.7 Dielectric Constant - MIL-P-27412 (USAF)
- 4.3.8 Determination of Beryllium Concentration -
- 4.3.9 Determination of Hydrazine Concentration -
- 4.3.10 Determination of Hydrazine and Water -
- 4.3.11 Determination of Carbopol-940 -

### 5.0 QUALITY ASSURANCE PROVISIONS

#### 5.1 Acceptance

(U) The fuel acceptance shall be based on the measurement of yield stress, density, and smoothness. These properties shall be measured in accordance with the procedures specified in Paragraph 4.0. Subsequent tests shall be conducted to determine the concentration of the ingredients listed in Paragraph 3.3.1.

#### 5.2 Sampling

(U) Samples shall be taken in a manner to avoid contamination of either sample or remaining product. Every precaution must be taken to prevent contaminating the product with air, carbon dioxide, or water. Sample containers must be clean, dry, and preflushed with dry gaseous nitrogen.

### 6.0 PREPARATION FOR SHIPMENT

#### 6.1 Packaging -

#### 6.2 Marking -

#### 6.3 Labeling -

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Report AFRPL-TR-66-230, Appendix A

### 7.0 NOTES

#### 7.1 Intended Use

(U) The criteria and limits specified in this document are intended for use to define Beryllizine propellant. These criteria shall provide the means of characterizing and controlling the fuel during its development stages so that comparisons can be made of samples used in laboratory and engineering investigations, and so that samples can be compared which are prepared by different agencies.

#### 7.2 Safety Precautions

7.2.1 Flammability -

7.2.2 Toxicity -

7.2.3 Spills

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**Report AFRPL-TR-66-230**

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Report AFRPL-TR-66-230

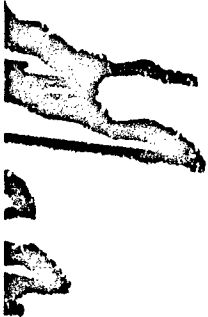
APPENDIX B

SURVEY OF VIBRATIONS AND ACCELERATION CONDITIONS

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Report AFRPL-TR-66-230



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Report AFRPL-TR-66-230, Appendix B

(U) The purpose of this survey was to accumulate and analyze the available data pertaining to the vibration and acceleration experienced by missile systems. From these data realistic tests to simulate these conditions can be developed for testing the mechanical stability of metalized propellants.

(U) At the start of this study it became apparent that the technology of measuring and defining environmental conditions for missile systems is in a state of evolution and the concepts are frequently subject to change and to different interpretations. A consequence is that in many cases only fragmentary data are reported for existing missile systems. This is partly because the telemetering and measuring techniques for the high-frequency vibrations experienced in missiles have not been developed to a level suitable for measuring the environments of airborne systems<sup>1</sup>. Most of the data on flight vibrations have been limited by the standard method of obtaining real-time vibration measurements about one axis at one vehicle location with the frequency response not going beyond 2,000 cps as limited by the utilization of the upper two sub-carriers of the standard FM telemeter<sup>2</sup>. Further problems arose from the test data being reported in a variety of terms. For example, vibrations are reported on missile systems expressed in terms of transmissibility, of frequency for single and double amplitudes, decibels, and spectral density ( $g^2/cps$ ). Coupled with the difference in terms of quantization is the fact that missiles encounter random vibrations. Consequently, any assessment of missile system environments for determining a general set of criteria is an extremely subjective operation, and such a set of criteria can only be practical if it is based on worse-condition situations.

(U) The data accumulated in this survey, reduced to comparable terms, are presented in Tables I and II and a short synopsis of the conclusions follows:

1. The maximum longitudinal acceleration experienced by liquid missile systems is 12 g.
2. The vibrations encountered by missiles in transportation and during flight are almost entirely random and have the following limiting characteristics:

---

\* Lawrence, H. C., "Shock and Vibration Design," Space/Aeronautics, p. 49 December 1961.

\*\* Clevenson, S. A., "Considerations of Vibration Environments in Space Flight Systems," in Proceed. of Inst. of Environ. Sci., p. 82, 1960

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix B

	<u>Frequency Bands, cps</u>	
	<u>5-500</u>	<u>500-2000</u>
<u>Acceleration Spectral Density,</u> $g^2/cps$	0.5	1.25
<u>Maximum Peak Acceleration, g rms</u>	5	50

The most significant measurements on systems were obtained from reports on Jupiter, Atlas, and Saturn in the Proceedings of the Institute of Environmental Sciences and on Titan II in The Acoustic and Vibration Environment of the Titan II Vehicle, Bolt, Beranek, and Newman, Inc., Cambridge, Mass., Report BSD-TDR-63-189, September 1963.

(U) Because the vibrations encountered by systems are random, the criterion of sinusoidal vibrations in many of the published specifications on components is artificial and meaningless with regard to providing the conditions for realistic failure, such as settling and separation in metalized propellants. Accordingly, a set of parameters should be used for testing propellant to random vibrations that is in accord with that described in the method by G. B. Booth, MB Electronics, "Sweep Random Vibration," p. 491, in Proceedings of the Institute of Environmental Sciences, 1960.

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Report AFRPL-TR-66-230, Appendix B

TABLE I  
VIBRATIONS ENCOUNTERED IN TRANSPORTING MISSILE SYSTEMS

Vibration		Peak Acceleration*, g	Type of Carrier	Meas. Data or Spec. Test	Reference/Source
Principal Frequency Range, cps	Mean				
1 - 200	0.5	5	Truck	Measured	Barbriere, R.E., WADC TR 58-363, ASTIA No. AD 204-095 (1958) quoted in AFSCM 80-9, Vol. V, p. B.1-14, Dec. 1964.
30 - 90	0.25	3	Rail	Measured	DOD Guided Missile Packaging Handbook, RD 219/3, July 1955, quoted in AFSCM 80-9, Vol. V, p. B.1-14, Dec. 1964.
2 - 20	0.2	0.3	Ship	Measured	<u>loc.cit.</u>
5 - 640	0.2	0.5	Jet Transport	Measured	AFSCM 80-9, Vol. V, p. B.1-17 and p. B.1-67, Dec. 1964.
5 - 50	1.5	4	Truck	Measured	Polaris Handling Manual, Aerojet-General Corp., Oct. 1963.
2 - 50	0.2	1.5	Rail	Measured	Polaris Handling Manual, Aerojet-General Corp., Oct. 1963.
1 - 50	4	10	Truck (rough road)	Measured	"Data from Pershing Motor Demonstration Program," SR 61073, Vol. II, Aerojet-General Corp., June 1961.
5 - 50	1.5	---	Truck	Spec.	MIL E-5272C (ASG) Environmental Testing

\* The vibrations reported are mostly random; however, in the sources the amplitudes are given in terms of sinusoidal vibrations.

# UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix B

TABLE I (cont.)

Principal Frequency Range, cps		Vibration Peak Acceleration*, g		Type of Carrier	Meas. Data or Spec. Test	Reference/Source
		Mean	Max.			
2 - 60 5 - 500	---	---	2	Pershing Missile Tracked Vehicle	Spec. values based on measured environment	Taravella, D.J., "Standardization of Nuclear Environmental Test Conditions," Picatinny Arsenal, in Proceedings of Inst. of Environ. Sci., 1963, p. 641.
			5			
10 - 500	0.5		1.5	Redstone Missile in Transporter; air, highway and field cond.	Measured	Nichols, R.P., "Combined Environmental Tests of Redstone Missile," p. 63 in Proceed. of Inst. of Environ Sci., 1959
0.5 - 40	---		3	Vehicle on Imp. Roads	Design Crit. for MMIREM	Letter to Dr. Simmons, Aerojet-General Corp., from E.W. Angelus, BSRPA, BSD, 26 March 1965.

\* The vibrations reported are mostly random; however, in the sources the amplitudes are given in terms of sinusoidal vibrations.

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Report AFRPL-TR-66-230, Appendix B

TABLE II  
INFLIGHT VIBRATIONS AND LONGITUDINAL ACCELERATION OF MISSILE AND PROPULSION SYSTEMS

Vibrations		Longitudinal Acceleration, g	System	Meas. Data or Spec. Req.		Reference
Principal Frequency Range, cps	Nominal Maximum Amplitude*					
60 - 2,000	1.25 g <sup>2</sup> /cps	---	Titan II	Measured		Bolt, Beranek, Newman, "The Acoustic and Vibrational Environment of the Titan II Vehicle," BSD-TDR-63-189, 1963.
5 - 2,000	0.4-in. DA	---	Gemini	Spec.		"GLVS Environmental Requirements and Tests," Martin Co., Spec. MB-1043.
20 - 3,000	1.5g (caused by aerodynamic noise, 153 db)	---	Mercury Capsule	Measured		Clevenson, S.A., "Vibration and Noise Environmental Studies for Project Mercury," p. 541, <u>Proceed. of Inst. of Environ. Sci., 1961.</u>
---	---	9	Mercury Atlas Booster	Measured		Chambers, R.M., "Human Engineering Studies in Acceleration Environments," p. 391 in <u>Proceed. of Inst. of Environ. Sci., 1964.</u>
0 - 2,000	8.5 g	---	ICBM Atlas	Measured		Clevenson, S.A., "Considerations of Vibration Environments in Space Flight Systems," NASA Langley Research Center, p. 87, <u>Proceed. of Inst., of Environ. Sci., 1960.</u>

\* Amplitudes are reported in terms of double amplitude, DA; acceleration level,  $\frac{1}{2}$  g; and acceleration spectral density, g<sup>2</sup>/cps. The latter is recognized as the most appropriate for describing random variations.

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Report AFRPL-TR-66-230, Appendix B

TABLE II (cont.)

Vibrations		Longitudinal Acceleration, g	System	Meas. Data or Spec. Req.	Reference
Principal Frequency Range, cps	Nominal Maximum Amplitude*				
0 - 2,000	17 g		IRBM Jupiter	Measured	<u>loc. cit.</u>
5 - 55	0.2-in. DA	7	Liquid Booster	Measured	"Environmental Engineering," AFSCM 80-9, Vol. V, p. B.1-19.
55 - 2,000	3 g	---	Sustained Flight	Measured	"Environmental Engineering," AFSCM 80-9, Vol. V, p. B.1-19.
10 - 2,000	1.25 g <sup>2</sup> /cps	---	Titan II	Measured	Harvey, W.L., "Specifying Vibrations Simulation," Mar. in Co., p. 407 in <u>Proceed. of Inst. of Env. ron. Sci., 1964;</u> Boss, R.E., "The Gemini Launch Vehicle Component Test Program," p. 597, <u>ibid.</u>
---	---	10.5	Aerobee	Measured	Roth, C.E., "Environmental Meas. in Sounding Rockets," p. 93, in <u>Proceed. of Inst. of Environ. Sci., 1961.</u>
5 - 2,000	0.2 g <sup>2</sup> /cps	---	Atlas	Measured Mercury Launch and Boost	Seat, R.L., "Environ. Testing of the Project Mercury Manned Satellite," McDonnell Aircraft Corp., p. 21 in <u>Proceed. of Inst. of Environ. Sci., 1962.</u>

\* Amplitudes are reported in terms of double amplitude, DA; acceleration level,  $\pm$  g; and acceleration spectral density, g<sup>2</sup>/cps. The latter is recognized as the most appropriate for describing random vibrations.

# UNCLASSIFIED

Report ATRPL-TR-66-230, Appendix B

TABLE II (cont.)

Vibrations		Longitudinal Acceleration, g	System	Meas. Data or Spec. Req.	Reference
Principal Frequency Range, cps	Nominal Maximum Amplitude*				
20 - 110	10 g	---	Saturn SA-1 1st Stage	Measured	Jewell, R.L., "Acoustic and Vibration Environment for Saturn," Chrysler Corp., p. 26, U.S.N. Res. Lab. Bull. No. 30, Part IV, <u>Shock</u> , Vibration and Assoc. Environ., April 1962.
10 - 1,000	3.5 g <sup>2</sup> /cps	---	Guided Missile Weapons	Design Requirement	MIL P-9024B (USAF), Specifica- tions and Design Require- ments for Guided Missile Weapon Systems.
40 - 2,000	1.0 g <sup>2</sup> /cps	7	Titan IILM	Predicted	"Titan Improvement Program," Final Report, CR-64-73, Martin Co., Denver, pp. III-8, III-68, June 1964. (Secret**)
20 - 60 60 - 500 500 - 2,000	9 g 5 g 3 g	---	Sparrow III	Spec.	Supp. No. 2, buWeps Spec. RMAI-001, 1 March 1960.
50 - 3,000	0.3 g <sup>2</sup> /cps	10	Transtage of Program 624A Launch Vehicle	Spec.	Aerojet Specification AGC- 10132C, Engine for Third Liquid-Propellant Stage, Program 624A.

\* Amplitudes are reported in terms of double amplitude, DA: acceleration level, + g; and acceleration spectral density, g<sup>2</sup>/cps. The latter is recognized as the most appropriate for describing random vibrations.

\*\* Cited data unclassified.

# UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix B

TABLE II (cont.)

Vibrations		Longitudinal Acceleration, g	System	Meas. Data or Spec. Req.	Reference
Principal Frequency Range, cps	Nominal Maximum Amplitude*				
(Diverse)	---	12	Ground- Launched	Spec.	MIL-STD-810A (USAF), 23 June 1964.
5 - 55 55 - 1,000 1,000 - 2,000	0.06-in. DA 10 g 20 g	8 - 10	Ballistic Systems	Antic. based on meas. envir.	Grimm, J., "A Glance at the Future," in Proceed. of Annual Meeting, Inst. of Environmental Engineers, 1957.
5 - 24 50 - 1,000 24 - 2,000	0.4-in. DA 0.1 g <sup>2</sup> /cps 12 g	12	FIRE Project, Component Atlas D Vehicle		Barber, W.J., "Project FIRE Velocity Package Environmental Test Program," p. 563, in Proceed. of Inst. of Environ. Sci., 1964.

\* Amplitudes are reported in terms of double amplitude, DA; acceleration level,  $\pm$  g; and acceleration spectral density,  $g^2$ /cps. The latter is recognized as the most appropriate for describing random vibrations.



**CONFIDENTIAL**

Report AFRPL-TR-66-230

APPENDIX C

TENTATIVE MILITARY SPECIFICATION  
PROPELLANT, BERYLLIZINE (U)

Page 189

**CONFIDENTIAL**

(This page is Unclassified)

**CONFIDENTIAL**

Report AFRPL-TR-66-230, Appendix C

Page 190

**CONFIDENTIAL**

(This page is Unclassified)

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## TABLE OF CONTENTS

		<u>Page</u>
(U)	1.0 SCOPE	193
	1.1 <u>Purpose</u>	193
	1.2 <u>Summary of Propellant Requirements</u>	193
	1.2.1 Propellant Property Requirements	193
	1.2.2 Quality Assurance Requirements	193
	1.2.3 Handling Requirements	193
(U)	2.0 APPLICABLE DOCUMENTS	193
	2.1 <u>Military Documents</u>	193
	2.2 <u>Other Publications</u>	194
(C)	3.0 PROPELLANT PROPERTY REQUIREMENTS	195
(U)	3.1 <u>Composition and Physical Properties</u>	195
(U)	3.2 <u>Filtering</u>	195
(C)	3.3 <u>Components Quality Requirements</u>	196
	3.3.1 Beryllium	196
	3.3.2 Gelling agent, Modified Polyacrylic Acid	197
	3.3.3 Hydrazine	197
(C)	4.0 QUALITY ASSURANCE REQUIREMENTS	198
	4.1 <u>Responsibility for Inspection</u>	198
	4.2 <u>Drum Inspection</u>	198
(U)	4.3 <u>Sampling</u>	198
(U)	4.4 <u>Classification of Tests</u>	199
(U)	4.5 <u>Test Conditions</u>	199
(U)	4.6 <u>Quality Conformance Tests</u>	199
(C)	4.7 <u>Test Methods - Components Quality</u>	200
	4.7.1 Beryllium	200
	4.7.2 Modified Polyacrylic Acid	201
	4.7.3 Hydrazine	201
(C)	4.8 <u>Test Methods - Propellant Composition and Properties</u>	201
	4.8.1 Beryllium Concentration	201
	4.8.2 Hydrazine and Water Concentrations	204
	4.8.3 Modified Polyacrylic Acid Concentration	208

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## Table of Contents (cont.)

		<u>Page</u>
	4.8.4 Density	210
	4.8.5 Yield Stress	213
	4.8.6 Characteristic Flow Curve	218
	4.8.7 Smoothness	222
(U)	4.9 <u>Rejection and Retest</u>	222
(U)	5.0 HANDLING REQUIREMENTS	223
(U)	5.1 <u>Packaging</u>	223
(U)	5.2 <u>Marking</u>	223
(U)	5.3 <u>Labeling</u>	223
(U)	6.0 NOTES	224
(U)	6.1 <u>Intended Use</u>	224
(U)	6.2 <u>Safety Precautions</u>	224

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## 1.0 SCOPE

### 1.1 Purpose

(C) The purpose of this document is to specify the propellant requirements for assuring the quality of the two gelled beryllium-hydrazine fuels designated Beryllizine-21 and Beryllizine-28. The specified properties and limits, the specified equipment and test procedures, and the other quality assurance provisions in this propellant specification will be revised as necessary.

### 1.2 Summary of Propellant Requirements

(U) The items that are specified as requirements for controlling the quality of the Beryllizine fuels are as follows:

#### 1.2.1 Propellant Property Requirements

Composition

Physical Properties

Component Quality

#### 1.2.2 Quality Assurance Requirements

Container Inspection

Sampling

Test Methods

#### 1.2.3 Handling Requirements

## 2.0 APPLICABLE DOCUMENTS

### 2.1 Military Documents

(U) The following documents, of the issue in effect on the date of invitation for bids or request for proposal, form a part of this document to the extent specified herein.

### SPECIFICATIONS

MIL-P-26536 Propellant, Hydrazine

MIL-P-27412 Propellant, Aluminum-Hydrazine Gelled

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 2.0, Applicable Documents (cont.)

MIL-P-27401 Propellant, Nitrogen, Pressurizing

MIL-P-27407 Propellant, Pressurizing Agent, Helium

### STANDARDS

MIL-STD-105 Sampling Procedures and Table for  
Inspection by Attributes

MIL-STD-172A Color Code for Containers of Liquid  
Propellants

MIL-STD-129 Marking for Shipment and Storage

MIL-STD-414 Sampling Procedures and Tables for  
Inspection by Variables for Percent  
Defective

(Copies of specifications, standards, drawings and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

### 2.2 Other Publications

(U) The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

### PUBLICATIONS

#### American Society for Testing and Materials

Method of Testing Viscosity, ASTM D-1092

#### Interstate Commerce Commission

49 CFR 71-90 Interstate Commerce Commission  
Regulations for Transportation of Explosives  
and Other Dangerous Articles

### SPECIFICATIONS

#### Interstate Commerce Commission

ICC-5C

ICC-103C-W

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## 2.0 Applicable Documents (cont.)

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington 25, D.C.)

## 3.0 PROPELLANT PROPERTY REQUIREMENTS

### 3.1 Composition and Physical Properties

(U) The composition and physical properties of the fuels shall be as specified in Table I when tested in accordance with the applicable test method.

TABLE I

<u>Requirements</u>	<u>Limit</u>		<u>Test Method</u>
	<u>Beryllizine-21</u>	<u>Beryllizine-28</u>	
<u>Composition</u>			
Beryllium, wt%	21.0 $\pm$ 0.5	28.0 $\pm$ 0.5	4.8.1
Hydrazine, wt %	77.5 min	70.5 min	4.8.2
Modified polyacrylic acid, wt %	0.55 - 0.60	0.50 - 0.60	4.8.3
Water, wt %	0.8 max	0.8 max	4.8.2
<u>Physical Properties (77°F)</u>			
Density, gm/cc	1.105 to 1.120	1.145 to 1.160	4.8.4
Yield Stress, dynes/cm <sup>2</sup>	1400 to 2000	1400 to 2000	4.8.5
Characteristic Flow Curve	per Figure 1	per Figure 1	4.8.6
Smoothness	Smooth	Smooth	4.8.7

(CONFIDENTIAL TABLE)

### 3.2 Filtering

(U) A filter screen having a one-eighth-inch mesh shall be installed between the supplier's plant system and the container to be filled for shipment.

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 3.0, Propellant Property Requirements (cont.)

#### 3.3 (C) Components Quality Requirements

##### 3.3.1 Beryllium

(U) The beryllium powder used in the propellants shall be in accordance with the following paragraphs.

##### 3.3.1.1 Composition

(C) The chemical composition of the beryllium powder shall be as specified in Table II.

TABLE II

#### BERYLLIUM POWDER COMPOSITION REQUIREMENTS (U)

<u>Requirements</u>	<u>Limit</u>	<u>Test Method</u>	<u>AQL</u>	<u>Inspection Level</u>
Free beryllium, wt %	- min			
Volatiles, wt %	-			
Ether Solubles, wt %	-			
Iron, wt %	-			
Particle size - -325 mesh %	-			
at 90% probability, microns	-			
at 50% probability microns	-			

(CONFIDENTIAL TABLE)

##### 3.3.1.2 Material treatment compliance

(C) The beryllium powder shall have been treated by the process of \_\_\_\_\_ and the test of effectiveness of treatment shall be \_\_\_\_\_.

##### 3.3.1.3 Soluble Electrolytes

(C) The beryllium powder shall not contain an amount of hydrazine-soluble electrolytes that would result in a dielectric



## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 3.0, Propellant Property Requirements (cont.)

constant of hydrazine in excess of \_\_\_\_\_  
when tested in accordance with \_\_\_\_\_.

#### 3.3.1.4 Particle Size Distribution

(U) The particle size distribution, in weight percent, shall be in accordance with Table II.

#### 3.3.1.5 Maximum Particle Size

(U) No particle shall have a dimension greater than 0.030 in. in accordance with \_\_\_\_\_.

#### 3.3.2 Gelling Agent, Modified Polyacrylic Acid

(U) The gelling agent used in the propellants shall be as follows.

##### 3.3.2.1 Material

(U) The material shall be a polyacrylic acid powder conforming to the properties in Table III when tested in accordance with the applicable test method specified.

TABLE III

#### MODIFIED POLYACRYLIC ACID PROPERTIES

<u>Requirement</u>	<u>Limit</u>	<u>Test Method</u>
Moisture, wt %	2.0 max	4.7.2.3
Heavy metals, ppm	10 max	4.7.2.4

(UNCLASSIFIED TABLE)

#### 3.3.3 Hydrazine

(U) The hydrazine used in the propellants shall be in accordance with MIL-P-26536 and as specified in para. 3.3.3 of MIL-P-27412, the latter specifying the following three requirements:

(a) The water concentration less soluble impurities shall not exceed 1% by weight.

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 3.0, Propellant Property Requirements (cont.)

(b) The hydrazine assay shall be a minimum of 98.5% by weight.

(c) A dielectric constant not exceeding 47 is a specified requirement.

### 4.0 QUALITY ASSURANCE REQUIREMENTS

#### 4.1 Responsibility for Inspection

(U) Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

#### 4.2 Drum Inspection

(U) Drums containing Beryllizine propellant shall be free from defects, leakage, and any indications of propellant decomposition when inspected in accordance with 4.2.1.

##### 4.2.1 Inspection of Filled Drums

(U) Each filled drum selected in accordance with 4.3.3 shall be examined for defects of the drum and the closure for evidence of leakage, and weighed to determine the amount of the contents for proper filling. Each selected drum shall be inspected to detect any unusual temperature or sign of any activity caused by the propellant.

#### 4.3 Sampling

##### 4.3.1 Lot

(U) A lot shall consist of the propellant produced by one manufacturer, with no change in process or materials, in not more than 24 consecutive hours provided the operation is continuous. In the event the process is a batch process, each batch shall constitute a lot.

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 4.0 Quality Assurance Requirements (cont.)

#### 4.3.2 Test Samples

(U) Samples shall be taken in such a manner as to avoid contamination of either sample or remaining product. Every precaution must be taken to prevent contamination of the product with air, carbon dioxide, or water. Sample containers must be clean, dry, and previously flushed with dry, carbon dioxide (CO<sub>2</sub>) free, nitrogen.

#### 4.3.3 Drums

(U) Unless otherwise specified, samples shall be drawn by means of an accepted sampling device from a number of drums selected at random and equal to the cube root (or the next higher whole number) of the total number of drums comprising a lot. The sample shall be taken approximately 1 in. below the surface of the propellant. If more than one lot is represented in a shipment, then each lot represented shall be considered as a separate shipment for sampling purposes.

#### 4.3.4 Drum Filling

(U) When specified by the contract or order (see 6.5), samples shall be taken during the process of filling drums from a storage tank. One sample shall be taken at the beginning, one when half the drums are filled and one while the last drum is being filled.

#### 4.3.5 Tanks

(U) Samples shall be taken by means of a sampling thief or other accepted device, from each tank truck, railroad tank car, or storage tank offered for delivery before drum filling. One sample shall be drawn within 1 ft of the surface, one near the mid-point, and one within 1 ft of the bottom.

### 4.4 Classification of Tests

(U) The inspection and testing of the propellant shall be classified as quality conformance tests.

### 4.5 Test Conditions

(U) The test conditions are described under the individual tests to which they apply.

### 4.6 Quality Conformance Tests

(U) Quality conformance tests shall consist of:

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## 4.0, Quality Assurance Requirements (cont.)

(a) Qualification tests . . . . . 4.6.1

(b) Acceptance tests . . . . . 4.6.2

### 4.6.1 Qualification Tests

(U) The propellant shall be subject to the following tests as described under 4.8 and components tests under 4.7.

### 4.6.2 Acceptance Tests

(C) The propellant shall be selected in accordance with 4.3 and subjected to the following tests as described under 4.8.

(a) Composition and physical properties . . . 4.8.1

(b) Beryllium . . . . . 4.8.1

(c) Hydrazine and water . . . . . 4.8.2

(d) Modified polyacrylic acid . . . . . 4.8.3

(e) Density . . . . . 4.8.4

(f) Yield Stress . . . . . 4.8.5

(g) Characteristic flow curve . . . . . 4.8.6

(h) Smoothness . . . . . 4.8.7

## 4.7 Test Methods - Components Quality

### 4.7.1 Beryllium

#### 4.7.1.1 Quality Conformance Tests

(U) Quality conformance testing of individual lots shall consist of the tests in Table II and particle size requirements in 4.5.1.4.

#### 4.7.1.2 Free Beryllium

(U) To be determined.

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## 4.0, Quality Assurance Requirements (cont.)

### 4.7.2 Modified Polyacrylic Acid

#### 4.7.2.1 Quality Conformance Tests

(U) Quality conformance testing shall be in accordance with Table III, and as qualified according to MIL-P-27412, paragraph 4.5.2.

#### 4.7.2.2 Sampling

(U) When specified by the contract or order, samples shall be taken during this process of filling drums. One sample shall be taken at the beginning, one when half the drums are filled, and one while the last drum is being filled.

#### 4.7.2.3 Moisture Content

(U) Qualified according to MIL-P-27412, para.

4.5.2.3.

#### 4.7.2.4 Heavy Metals

(U) Qualified according to MIL-P-27412, para.

4.5.2.4.

### 4.7.3 Hydrazine

(U) Hydrazine shall be tested in accordance with MIL-P-26536, and paragraph 3.3.3 of this specification. The dielectric constant shall be tested according to the method specified in para. 4.5.3.1 in MIL-P-27412.

## 4.8 Test Methods - Propellant Composition and Properties

(U) The test methods specified in Table I shall be used for the determination of the composition and physical properties. Reagents required shall be prepared from reagent grade chemicals.

### 4.8.1 Beryllium Concentration

(C) This method is based on the titrimetric measure of hydroxide ions (OH) liberated by the action of sodium fluoride on precipitated beryllium hydroxide. This method is applicable to materials that are known not to contain aluminum, zirconium, hafnium and rare earth elements, which can interfere. Triplicate tests shall be made on each propellant sample. The following reagents and equipment are required:

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

## 4.0, Quality Assurance Requirements (cont.)

### (a) Reagents

- (1) Sodium hydroxide, 50% solution
- (2) Hydrochloric acid, standard solution, 0.5N
- (3) Hydrochloric acid solution, 6N
- (4) Sodium fluoride powder, (J.T. Baker reagent, Catalog No. 3688)
- (5) Acetone, reagent grade

### (b) Equipment

- (1) pH meter, sensitivity,  $\pm 0.05$  pH unit
- (2) Magnetic stirrer
- (3) Polyethylene washing bottle, 125 ml, with PE tubing (Braun-Knecht-Heimann Co., Catalog 63, No. 16651)
- (4) Dry box, nitrogen-filled, for handling toxic material
- (5) Polyethylene tube, 1/4-in. ID, 4-in. long
- (6) Berzelius beaker, 300 ml
- (7) Volumetric pipet, Kimax, 25 ml (Braun-Knecht-Heimann Co., Catalog 63, No. 53045)

#### 4.8.1.1 Procedure

(U) (a) Weigh to the nearest 0.1 mg the Berzelius beaker containing about 50 ml of distilled water. Inside a nitrogen-filled drybox, draw the propellant into a 1/4-in. ID polyethylene tube. Then expel approximately 5.0 gm of sample out of the tubing into the 300-ml, weighed Berzelius beaker containing water, and weigh to the nearest 0.1 mg. Use a glass stirring rod to disperse the propellant sample into the water and mix in 20 ml of acetone. Then place 50 ml of 6N HCl in the specified polyethylene washing bottle, and slowly add 2-ml portions into the test mixture while stirring. The addition time of the total 50 ml of acid depends on the ebullience of any reactions. Caution: This reaction may be vigorous and the evolved gases might produce foam that could froth out of the beaker.

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

(U) (b) After all reaction subsides, wash down the inside walls of the beaker several times with distilled water so that all particles of metal are in contact with the acid solution. To ensure that the sample is completely dissolved, gently boil the mixture for 20 min and then cool to room temperature. Transfer the solution into a 250-ml volumetric flask and dilute to the reference mark.

(U) (c) With a pipet transfer a 25.00-ml aliquot of the test mixture into a 250-ml beaker containing a Teflon-covered magnetic stirring bar. Add sufficient quantity of distilled water to facilitate coverage of the pH probes. Immerse the pH meter probes and add dropwise with stirring a solution of 50% sodium hydroxide until a precipitate appears. Continue stirring until the precipitate dissolves. Continue the dropwise addition of the 50% sodium hydroxide solution with constant stirring until a permanent precipitate forms and the pH meter indicates a pH within the range from 10.5 to 11.0.

(U) (d) Allow the basic mixture to stand for a minimum time of 10 min (standing time is important), then carefully titrate with the standard 0.5N hydrochloric acid to the pH of 8.5 (+0.05). The end point is attained when no changes occur for 30 sec following the last addition of acid. A white precipitate of beryllium hydroxide will be present.

(U) (e) Add 8 gm (weighed to the nearest 0.5 gm) of sodium fluoride powder to the test mixture with stirring. Allow to stand for 30 min, then titrate the mixture with the standard 0.5N hydrochloric acid to a pH of 8.5 (+0.05), as described in step (d). Record the volume (ml) of standard 0.5N HCl required to reach the end point.

(C) (f) Calculations. The percent of total beryllium of the propellant sample is calculated as follows:

where,

N, equivalents/liter = normality of standardized 0.5N HCl

Vol Std Acid, ml = volume of standard 0.5N HCl required to titrate fluoride-liberated base

4.506 = beryllium equivalent weight, gm

$$\% \text{ Be} = \frac{(\text{Vol Std Acid, ml}) \times N \times 4.506 \times 10}{\text{propellant sample wt, gm}}$$

(C) The arithmetic average of the results from triplicate tests shall be reported in three significant figures as the beryllium concentration in the propellant.

## CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

#### 4.8.2 Hydrazine and Water Concentrations

The concentrations of these two constituents are measured with a gas chromatograph.

##### 4.8.2.1 (U) Required equipment and materials.

- (a) Gas chromatograph: incorporating a thermal conductivity detector.
- (b) Recorder: potentiometric strip chart 0-1 millivolt with integrator.
- (c) Tubing: stainless steel or aluminum, 1/4-in. x 40-in. long with 0.035-in. wall thickness.
- (d) Teflon VI powder: 60-70 mesh.
- (e) Carbowax 600: polyethylene glycol.
- (f) Methylene dichloride: ACS reagent grade.
- (g) Hypodermic syringe: 10 microliter, cemented needle.
- (h) Hypodermic syringe: 100 microliter, cemented needle.
- (i) Cylinder: helium gas, conforming to MIL-P-27407.
- (j) Regulator: helium, to fit the cylinder.
- (k) n-butanol: spectrographic grade.
- (l) sodium chloride: ACS reagent grade.

##### 4.8.2.2 Chromatograph Column Preparation

(U) Dissolve 5 gm of Carbowax 600 (polyethylene glycol 600) in 50 ml of methylene dichloride contained in a 400-ml beaker. While stirring, add 100 gm of powdered Teflon VI to the beaker. Allow the mixture to stand for 2 to 5 min, alternately stirring and tapping to ensure that all the solid support is coated with the liquid phase. Complete the



## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

evaporation of the liquid by spreading the material in a thin layer in a tray or casserole. Fill a 1/4-in. x 40-in. stainless steel or aluminum tubing, pouring the prepared material through a small funnel attached to one end. The bottom of the tubing is plugged with a small wad of glass wool. Tap gently or use a mechanical vibrator to facilitate packing. When the column is full, plug the inlet with glass wool and bend or coil the tubing as required and connect to the gas chromatograph.

### 4.8.2.3 Calibration and Peak Area Factors

#### (a) Instrument Calibration

(U) To about 20 gm of Beryllizine of known composition (except for water) in tared, glass-stoppered, 50-ml centrifuge tubes, add known quantities of water to the samples to give a range of water concentrations up to 2% by weight. The hydrazine contents shall then be corrected for the new total weights. To these standard mixtures, add dry n-butanol as an internal standard in the amount of  $32.0 \pm 0.1\%$  based on the final weight. Add 5-6 mg of dried, finely-powdered NaCl and thoroughly shake the tubes until the aluminum is free-flowing. Centrifuge each sample mixture to produce a clear supernatant. Inject a 2-microliter portion of the supernatant into the chromatograph. Under the instrumental conditions outlined in 4.8.2.4 the hydrazine peak will be produced on the recorder paper approximately 15 min after injection. Measure the areas of the hydrazine, water and butanol peaks, and record these data, respectively, as  $A_{h-kn}$ ,  $A_{w-kn}$ , and  $A_{but-kn}$ .

#### (b) Determine the $K_h$ Factor for Hydrazine

(U) The  $K_h$  factor for hydrazine is calculated from the measured peak areas mentioned above obtained from testing the sample of known concentrations of hydrazine and butanol (internal standard). The factor is obtained with the following equation:

$$K_h = \frac{(A_{but-kn}) \times (\text{percent } N_2H_4 \text{ in known sample})}{(A_{h-kn}) \times (\text{percent butanol in known sample})}$$

This factor is recorded for use in tests on samples of unknown hydrazine concentration.

#### (c) Determine the $K_w$ Factor for Water

(U) Before the  $K_w$  factor can be calculated from the measurements on the known samples, the percent water in the samples must be established. The percent water includes the known water added to the

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

samples and the water originally present. Obtain the percentage of original water by plotting the ratios of water peak area to butanol peak area against the percentages of added water in the several samples. The original water concentration of the Beryllizine is obtained by extrapolating the curve to zero on the ratio ordinate. The total water concentration is then determined for the samples included in the calculation testing. With the measured peak areas and concentrations of water and butanol (internal standard) for the tested calibration samples, the  $K_w$  factor for water shall be determined with the following equation:

$$K_w = \frac{(A_{\text{but-kn}}) \times \text{percent H}_2\text{O in known sample}}{(A_{\text{w-kn}}) \times (\text{percent butanol in known sample})}$$

This factor is recorded for use on samples of unknown water concentration.

#### 4.8.2.4 Test Procedure for Propellant Samples

##### (a) Chromatograph Preparation

(U) The column is prepared under para. 4.8.2.2 above. Adjust the helium flow rate between 60-120 cc/min. When the helium inlet pressure is below 10 psig or above 30 psig, the column is packed either too loose or tight and shall be repacked. Set the oven temperature at 90°C. When the gas chromatograph has separate heating controls for the injector and the detector, set both at 90°C. Condition the column by alternately injecting 100-microliter and 5-microliter samples of hydrazine until the minimum tailing and maximum signal is obtained for the hydrazine peak. The column will be conditioned prior to each analysis or series of analyses. If more than 20 min elapse between the elution of hydrazine and the injection of a new sample, recondition the column.

##### (b) Sample Testing

(U) In a dry box, transfer approximately 20 gm of Beryllizine to a tared, glass-stoppered, 50-ml centrifuge tube. (The glass-stopper is lubricated with silicone grease.) Stopper, remove from the dry box and reweigh to obtain the weight of sample. Quickly unstopper the tube and add dry n-butanol as an internal standard in the amount of 32.0 ± 0.1% based on the final weight and reweigh. Add 5-6 mg of dried, finely powdered NaCl and thoroughly shake the tube until the mixture is free-flowing. Centrifuge the mixture for at least 10 min at 1200 rpm to obtain a clear, liquid layer. Use a clean, dry, 10-microliter hypodermic syringe and draw up 8 microliters of sample. Invert the syringe and expel the air bubbles. Carefully set the syringe plunger to the 2-microliter mark and wipe the tip with a piece of tissue without touching the open end of the needle. Pierce

## UNCLASSIFIED

Report AFRPL-TR-660230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

the system of the gas chromatograph injection port and quickly expel the sample completely into the instrument. Withdraw the empty syringe instantly. Measure the areas of the hydrazine, water, and butanol peaks on the chart paper, and record these data as  $A_{h-unkn}$ ,  $A_{w-unkn}$ , and  $A_{but-unkn}$ , respectively.

The known concentration of butanol (the internal standard) must also be recorded with the data.

#### 4.8.2.5 Calculations of hydrazine and water concentrations in propellant samples

##### (a) Hydrazine concentration

(U) The area of the hydrazine peak measured on the unknown sample,  $A_{h-unkn}$ , is used with (1) the hydrazine peak area factor,  $K_h$ , (2) the area of the butanol peak measured on the unknown sample, and (3) the known percent concentration of butanol (internal standard) added to the unknown sample. These data are used to calculate the hydrazine concentration with the following equation:

$$\% \text{ hydrazine} = \frac{(A_{h-unkn}) \times (\text{percent butanol}) \times K_h}{(A_{but-unkn})}$$

Duplicate determinations shall be performed on all samples, and when two results on a sample differ by 1.0, the propellant sample shall be retested until duplicate analyses agree within the spread of 1.0. The arithmetic mean of the two results shall be reported to the nearest 0.1%. The accuracy of this method for determining hydrazine concentrations is  $\pm 0.5\%$ .

##### (b) Water Concentration

(U) This concentration is determined in manner similar to that for hydrazine concentration. The area of the water peak measured on the unknown sample,  $A_{w-unkn}$ , is used with (1) the water peak area factor,  $K_w$ , (2) the area of the butanol peak measured on the unknown sample, and (3) the known percent concentration of butanol (internal standard) added to the unknown sample. These data are used to calculate the water concentration with the following equation:

$$\% \text{ water} = \frac{(A_{w-unkn}) \times (\text{percent butanol}) \times K_w}{(A_{but-unkn})}$$

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

Duplicate determinations shall be performed on all samples, and when two results have a spread greater than 0.2%, the propellant sample shall be retested until duplicate analyses agree within that limit. The arithmetic mean of the two results shall be reported to the nearest 0.05% as the water concentration in the propellant. The accuracy of this water determination is  $\pm 0.1\%$ .

#### 4.8.3 Modified Polyacrylic Acid Concentration

(U) The concentration of the gelling agent in a propellant sample is determined by a gravimetric procedure. A sample of propellant is mixed with water, and the metal component is dissolved by acid. The gelling agent is precipitated by the acid and is isolated and measured gravimetrically.

# UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

## 4.0, Quality Assurance Requirements (cont.)

### 4.8.3.1 Required equipment and materials.

- (a) Analytical balance, sensitivity,  $\pm 1$  mg
- (b) 100-ml beaker
- (c) steam bath
- (d) filtering crucible, Coors, 30 ml
- (e) Drying oven
- (f) Muffle furnace
- (g) Hydrochloric acid 6N

### 4.8.3.2 Procedure.

(U) (a) Run triplicate tests. Weigh an 8- to 10-gm sample of propellant to the nearest 10 mg into a tared 100-ml beaker. Transfer the sample into an 800- to 1000-ml beaker by rinsing with about 50 ml of distilled water.

(U) (b) Cautiously add approximately 200 ml of 6N hydrochloric acid to the mixture with constant stirring over a period of about 30 min. The slow addition of acid prevents loss by excessive foaming due to hydrogen gas liberation. Distilled water may be poured into the foam to prevent loss and to cool the solution. When the reaction subsides, add an additional 200 ml of 6N HCl, and place the mixture on a steam bath.

(U) (c) Allow the mixture to stay on the steam bath until all of the beryllium is dissolved, and only the white flocculent solid remains in suspension along with any undissolved beryllium oxide (BeO).

(U) (d) Filter the solution (with vacuum) through a tared, 30-ml, porous-bottom Coors crucible fitted with a glass filter. Filter the liquid portion first, because the gelling agent will clog the filter and make subsequent filtering impossible.

(U) (e) Place the crucible in a drying oven at 212°F (100°C), and dry the contents of the crucible to a constant weight. The material in the crucible may have a grey color due to the presence of beryllium oxide. Record the weight as the weight of the gelling agent and beryllium oxide.

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

(U) (f) Place the crucible in a furnace at 800°F (427°C) for several hours to air-oxidize the agent to volatile products. Reweigh the cooled crucible to determine the weight of beryllium oxide remaining.

#### 4.8.3.3 Calculations

(U) Calculate the modified polyacrylic acid concentration with the following equation:

$$\% \text{ modified polyacrylic acid} = \frac{(\text{Wt of gelling agent} + \text{BeO}) - (\text{Wt BeO}), \text{ gm} \times 100}{\text{propellant sample, gm}}$$

The accuracy of the method is about 0.02 %. The results from the triplicate measurements shall be averaged arithmetically, and the average shall be reported to the nearest 0.01 %.

#### 4.8.4 Density

(U) The density of the propellant shall be measured by a pycnometric technique utilizing a layer of mineral oil to adjust to a standard volume. Mineral oil is added to a weighed sample of propellant in a ground-glass stoppered, 25-ml volumetric flask to adjust the volume to the calibration mark. The standard volume of the flask, less the volume occupied by the propellant sample, is the volume used to calculate the sample density. The precision and uncertainty among sample measurements is 0.003 gm/cc. Determine the density of the propellant in accordance with 4.8.4.1 through 4.8.4.4 with the following required equipment:

(U) (a) Flask, volumetric, 25 ml, calibrated at 77°F, (25°C) with standard ground-glass stoppers, 3 required.

(U) (b) Water bath, temperature regulated at 77° ± 0.2°F (25°C).

(U) (c) Analytical balance, sensitivity 0.5 mg (a rapid-weighing Mettler or Sartorius balance is recommended).

(U) (d) Eye droppers, common laboratory type.

(U) (e) Mineral oil, accurately measured density at 77°F (25°C), one quart required. Nujol, by Plough, Inc. Los Angeles, Calif.

## UNCLASSIFIED

### Report AFRPL-TR-66-230, Appendix C

#### 4.0, Quality Assurance Requirements (cont.)

##### 4.8.4.1 Preparation.

(U) (a) The 25-ml volumetric (density) flasks shall be thoroughly cleaned and dried before use, including new flasks. The standard chromic acid cleaning solution is recommended, followed by five distilled water rinses, two rinses with methanol, and drying in an oven or with a stream of air or gaseous nitrogen.

(U) (b) Immediately prior to use, number the three density flasks and stoppers, purge with gaseous nitrogen and stopper each. Lubricant shall not be used with the glass stopper.

##### 4.8.4.2 Procedure.

(U) (a) Weigh the empty, stoppered density flasks on an analytical balance and record the weight of each flask as the tare weight,  $W_{T_0}$ .

(U) (b) Place the propellant sample in the bottom of the density flask with a pipette, or allow to flow in under pressure through a 1/4-in. stainless steel tube inserted down the neck of the flask. The sample size shall be sufficient to fill about three-fourths of the body of the flask. The recommended technique for admitting the sample into the flask is to extend the tube close to the bottom, and allow material to slowly flow out, and carefully raise the tube so the tube end is constantly at the surface of the material in the flask. This technique helps to minimize entrapped bubbles in the sample and it ensures that the outside of the delivery tube is not covered with sample material that can rub off onto the neck of the density flask, and contribute to a volume error. When material accidentally contacts the neck of the flask, remove material with a cotton-tipped swab. Stopper the density flasks immediately after filling.

(U) (c) Weigh the density flasks containing samples and record the weights as tare plus sample weight,  $W_{T_S}$ .

(U) (d) Add mineral oil on top of the sample with an eye dropper to a level about 5 mm beneath the calibration mark, and stopper the flask. Wipe the outside of the eye dropper with a paper tissue each time before it is inserted into the neck of the flask to prevent oil from rubbing off the dropper onto the neck of the flask above the calibration mark. Immerse the density flasks to the level of the calibration mark in a water bath regulated at  $77 \pm 0.2^\circ\text{F}$  ( $25 \pm 0.1^\circ\text{C}$ ).

(U) (e) After 2 hrs, remove the flasks from the water bath and, with an eye dropper, immediately add mineral oil dropwise to the contents to adjust the level to the calibration mark. Stopper the flask.

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

(U) (f) Weigh the density flasks for the third time, and record the weight of the flask plus sample plus mineral oil as  $Wt_{TSO}$ .

#### 4.8.4.3 Cleaning.

(U) Purge the density flasks with water through a 1/4-in. tube inserted through the flask neck. The flushing water will carry the bulk of the sample out of the flask, and the residue can be easily removed after adding a 1% salt water solution to the flask to break the gel.

Caution: Waste propellant samples shall be combined with at least 10 volumes of water and always stored in an open container prior to disposal.

#### 4.8.4.4 Calculations

(U) (a) Calculate the density of the propellant samples with the following equations:

$$\text{Volume of mineral oil, cc} = \frac{Wt_{TSO} - Wt_{TS}}{d_o}$$

$$\text{Sample density, gm/cc} = \frac{(Wt_{TS} - Wt_T)}{(25.00 - \text{Vol of min oil})}$$

Where:  $Wt_T$ , gm = Tare weight of density flask.

$Wt_{TS}$ , gm = Weight of density flask plus sample.

$Wt_{TSO}$ , gm = Weight of density flask plus sample plus mineral oil.

$d_o$ , gm/cc = Density 77°F (25°C) of mineral oil

(U) (b) Calculate sample densities to four significant figures. Report the arithmetic mean of the three sample densities as the measured density, 77°F (25°C), gm/cc.

(U) (c) The precision of the three measurements (average deviation from the mean) is obtained by totaling the difference of each from the arithmetic mean and dividing the sum by three. The precision shall be affixed within parentheses to the reported measured density.



## UNCLASSIFIED

### Report AFRPL-TR-66-230, Appendix C

#### 4.0, Quality Assurance Requirements (cont.)

##### 4.8.5 Yield Stress

(U) Determine the yield stress of the propellant sample using the rising sphere rheometer. A stainless steel sphere is used to strain a gel sample to the limit at which the gel structure yields and the flow condition commences. The strain force applied from the surface of the sphere is measured by a strain gage,<sup>2</sup> and the output is recorded on a strip chart. The yield stress, in dynes/cm<sup>2</sup>, is that stress just required to overcome viscoelastic resistance of the gel to flow. Perform the yield stress determination in accordance with 4.8.5.3 through 4.8.5.6 with the following required equipment and materials:

(U) (a) Standard sphere: 0.750-in. diameter, 347 SS, attached to a 12-in. stainless steel wire (0.010-in. diameter).

(U) (b) Strain gage: Statham Model G1-4-250, 113-gm (4-oz) capacity; Statham Instruments Inc., Los Angeles, California.

(U) (c) Tensile strength apparatus: Gardner Laboratory, Inc., Bethesda, Maryland.

(U) (d) Variable speed motor: Model GT 21 and controller; G. K. Heller Company, Las Vegas, Nevada.

(U) (e) Transducer-amplifier indicator: Sanborn Model 311; Sanborn Company, Waltham, Massachusetts.

(U) (f) Graphic Recorder: Varian Model G-10; Varian Associates, Palo Alto, California.

(U) (g) Vibradamp support plate: Fisher Scientific Company, Chicago, Illinois.

##### 4.8.5.1 Special assembly instructions.

(U) (a) To attach the support wire to the sphere, drill a hole through the mid-section of the sphere, and fix the stainless steel wire into position by silver soldering or by cementing with a suitable epoxy resin, followed by polishing the surfaces marred by the operation.

(U) (b) Connect a 100:1 voltage divider to the signal output of the transducer amplifier to provide a proper signal level to drive the graphic recorder and to provide proper damping of the recorder.

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

(U) (c) Use a Helipot calibrated resistor (0 to 90 scale units) with a locking level to replace the 0 to 10 unit dial resistor on the motor controller to provide reproducible and accurate motor-speed settings.

(U) (d) Use a three-way toggle switch (UP-OFF-DOWN) to replace the forward-reverse motor switch on the motor controller.

(U) (e) Connect an ON-OFF switch in the power line to the tensile-strength apparatus to shut off all power to the apparatus and the motor controller.

(U) (f) Fasten with screws a 2 x 10-in. aluminum plate to the column head of the tensile-strength apparatus to serve as the mounting arm for the strain gage.

(U) (g) Construct a small-V-shaped hanger with a slot in the "V" from a 3 x 1/4 x 1/16-in. aluminum strap and attach to the shaft of the strain gage to hold the sphere wire. All sphere wires shall have an epoxy bead on one end to slip into the slotted hanger.

(U) (h) Measure the rise rate of the head of the tensile-strength apparatus at various settings on the Helipot motor-speed dial. Determine the setting that produces a movement rate of  $2.7 \times 10^{-3}$  cm/sec for the column head and, hence, for the sphere. Special test items required are as follows:

(U) (1) Water bath: Constant temperature regulated at  $77 \pm 0.1^\circ\text{F}$  ( $25.0 \pm 0.2^\circ\text{C}$ ).

(U) (2) Laboratory jack: To support sample bottle.

(U) (3) Sample bottles: At least 2.75-in. diameter and 5-in. height, 450-ml sample capacity, wide mouthed, and extra lids prepared with 3/16-in. diameter hole in center.

(U) (4) Sealing compound: Plastic, Duxseal; Johns-Manville Company.

#### 4.8.5.2 Transducer-amplifier balancing preparation.

(U) (a) For optimum stability, warm-up for a period of 1 to 2 hr before final balancing and operation. Set the controls

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

for the initial balancing by setting the attenuator to OFF, the zero suppression switch to OUT, and rotate the sensitivity control fully CLOCKWISE.

(U) (b) Set the USE-BAL switch to USE, adjust the meter pointer to CENTER with the position control, and reset the USE-BAL switch to BAL.

(U) (c) Check for no load on the transducer. Rotate the attenuator CLOCKWISE until the meter deflects near right edge of scale. (Avoid stopping on Mech Zero during attenuator switching.) Adjust the pointer to a null with the C BAL control and set pointer to ZERO with R BAL control. (Note: The high-torque potentiometers are to prevent accidental unbalance.) At each attenuator setting, achieve a null with the C BAL control and pointer zero with R BAL control. If null point moves off scale, return the null to scale by adjusting sensitivity control.

(U) (d) Set the USE-BAL switch to USE, and perform a final adjustment of the R BAL control so the meter pointer does not move when the attenuator is changed from XI to X200 setting.

#### 4.8.5.3 Calibration of system.

(U) (a) Set both the recorder power switch and the servo unit switch to ON.

(U) (b) Set the attenuator switch to X100. At X100, the range of readout by the recorder is zero to 50 gm. Each chart unit is equal to 0.5 gm of strain force.

(U) (c) Adjust the zero-adj knob and set the recorder stylus to the zero line on the chart.

(U) (d) Suspend from the strain gage, a calibration weight (exact to the nearest 0.01 gm and close to 50 gm), such as two 20 gm and one 5 gm analytical weights combined with an accurately weighed wire and small alligator clip. Adjust the Sanborn sensitivity control and set the recorder stylus to the proper chart reading for the weight.

(U) (e) Calibrate the middle region of the scale by removing one 20 gm weight and the 5 gm weight from the calibration weight on the strain gage, and adjust the sensitivity control to obtain accurate readout.

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

#### 4.8.5.4 Sample preparation and sphere positioning.

(U) (a) With a gaseous nitrogen atmosphere in the sample bottles, fill sample bottles to approximately three-fourths full with sample material.

(U) (b) Uncap the sample bottle and place a clean sphere (at near room temperature) in the sample. If the gel structure is too great to allow the sphere to drop into a position in the lower one third of the sample, position the sphere by rapping the bottom of the bottle with the palm of the hand.

(U) (c) Pass the bead-end of the support wire through the 3/16-in. hole in the center of one of the extra lids for the sample bottle, and screw the lid onto the bottle to provide a non-air-tight cover. Temporarily seal the hole in the lid with a small piece of plastic sealing compound to provide a vapor-tight seal. The time that the propellant sample is open and exposed to air shall be kept to absolute minimum, less than 10 sec.

(U) (d) Place sample bottle with sphere in a constant temperature bath and allow contents to reach specified temperature of 77°F (25°C). The time for temperature conditioning shall not be less than 2 hr. The sample and sphere may be temperature conditioned separately. Place the sphere in the sample according to (a) and (b).

#### 4.8.5.5 Procedure.

4.8.5.5.1 (U) Connecting sphere-support wire to strain gage. After the sample and sphere have reached the desired temperature, place the sample bottle on the laboratory jack under the strain gage. Remove the plastic sealing compound from the center hold in the lid of the bottle, and examine the wire for freedom of movement in the hole. Slip the epoxy bead on the end of the support wire of the sphere into the slot in the hanger attached to the strain gage.

4.8.5.5.2 (U) Locating sphere position. Adjust the laboratory jack to position the sample bottle so the sphere is located at least one sphere-diameter from the bottom of the sample bottle. This position can be determined by previously marking the column of the tensile-strength apparatus at the level equal to the bottom of the suspended sphere. Examine the wire and verify the wire does not contact the sides of the hole in the lid. Kinks or bends in the wire do not affect the measurement of strain force, because the force is directly transmitted from the sphere regardless of slight bends in the support wire.

## UNCLASSIFIED

### Report AFRPL-TR-66-230, Appendix C

#### 4.0, Quality Assurance Requirements (cont.)

##### 4.8.5.5.3 Measuring strain force.

(U) (a) At the start of the test, the sphere will be straining the gel either slightly upward or downward, depending on the exact energy state the gel initially possesses in the region of the sphere. It is improbable that the sphere can be placed in the gel and wire attached to the strain gage and still leave the region surrounding the sphere undisturbed relative to state-of-strain. The state-of-strain of gel around the sphere will not affect the measurement of force required to strain the gel to its plastic limit. If the test is started with the gel already under a slight upward strain, the recorder stylus will remain at zero until the output of the strain gage is sufficient to produce a positive-upward strain force read-out. Tests have indicated that the final measurement of strain force at the elastic limit of the gel is not influenced by the slight gel strain at the start of the test.

(U) (b) Lock the motor-speed dial at the setting giving a sphere rise-rate of  $2.7 \times 10^{-3}$  cm/sec. Set the chart and motor switches to ON. When the curve of strain force drawn on the chart reaches a constant plateau for a period of two minutes, set the chart and motor switches to OFF to end the test. The time required is about 15 min.. Record the chart reading of strain force in the plateau region in chart units to the nearest one-half unit. One chart unit equals 0.5 gm. With this relationship, the chart reading is converted to grams and recorded as the maximum strain force, F.

##### 4.8.5.5.4 Shutdown.

(U) The power switch to the transducer-amplifier indicator shall remain in ON position when the rheometer is in continuous daily use. The power and servo switches to the graphic recorder and the power switch to the tensile-strength apparatus shall be in the OFF position during non-working hours.

##### 4.8.5.5.5 Cleanup and sample disposal.

(U) (a) Remove the sphere from the sample and flush with copious amounts of water to remove sample residue before rinsing with methanol and drying with a stream of compressed air or gaseous nitrogen. Clean and dry spheres shall be stored in a clean and capped glass bottle.

## UNCLASSIFIED

### Report AFRPL-TR-66-230, Appendix C

#### 4.0, Quality Assurance Requirements (cont.)

(U) (b) The proper lid to provide air-tight closure shall be screwed onto the bottle if the test sample is to be stored. If the sample is to be disposed, it should be combined with at least 10 volumes of water and stored in an open container to await further treatment.

#### 4.8.5.5.6 Calculations.

(U) The maximum strain force,  $F$ , in grams, shall be used in the calculation of yield stress. The "corrected weight" of the sphere is calculated by subtracting the buoyancy effect of the sample on the sphere from the weight of the sphere in air.

$$\text{Corrected weight, gm} = (\text{Wt of sphere, gm}) - (\text{Vol of sphere, cm}^3 \times \text{density of sample, gm/cm}^3)$$

$$\text{Yield stress, dynes/cm}^2 = \frac{(F - \text{Corr Wt}) \text{ gm} \times 980 \text{ dynes/gm}}{\text{Surface area of sphere, cm}^2}$$

#### 4.8.6 Characteristic Flow Curve

(U) This rheological property of the propellant is the curve constructed from measurements of flow rate and pressure drop through capillaries. The curve shows the relationship between shearing stress,  $D\Delta P/4L$ , psi, and shear rate,  $8V/D$ ,  $\text{sec}^{-1}$ . In these expressions  $D$  is capillary diameter,  $\Delta P$  is pressure drop,  $L$  is tube length, and  $V$  is mean linear velocity. Measurements on propellant shall be made with the ASTM D-1092 capillary viscometer, and a minimum of 10 data points at approximately distributed shear rates encompassing the shear-rate regime of 10 to 10,000  $\text{sec}^{-1}$  shall be used to construct the plot on the standard figure (Figure 1). Testing shall be conducted in accordance with 4.8.6.1 through 4.8.6.4.

##### 4.8.6.1 Required Equipment and Materials

(U) (a) ASTM D-1092 Capillary Viscometer

(U) (b) Viscometer capillaries:  $L/D = 40:1$ ,  
 $D \geq 0.050$  in. and  $L/D = 200$ ;  $D = 0.020$  in., 0.060 in., 0.140 in.

(U) (c) Hydraulic oil,  $150 \pm 30$  cp ( $77^\circ\text{F}$ ).  
(Duo Seal Pump Oil, The Welch Scientific Co., is suitable)

(U) (d) Thermocouple, 2 in., shielded end

(U) (e) Constant-temperature bath,  $77 \pm 0.2^\circ\text{F}$   
( $25 \pm 0.1^\circ\text{C}$ )

(U) (f) Copper tubing, 1/4-in. dia., 5-ft long, shaped into a thermostating coil fitted to the viscometer chamber.

UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

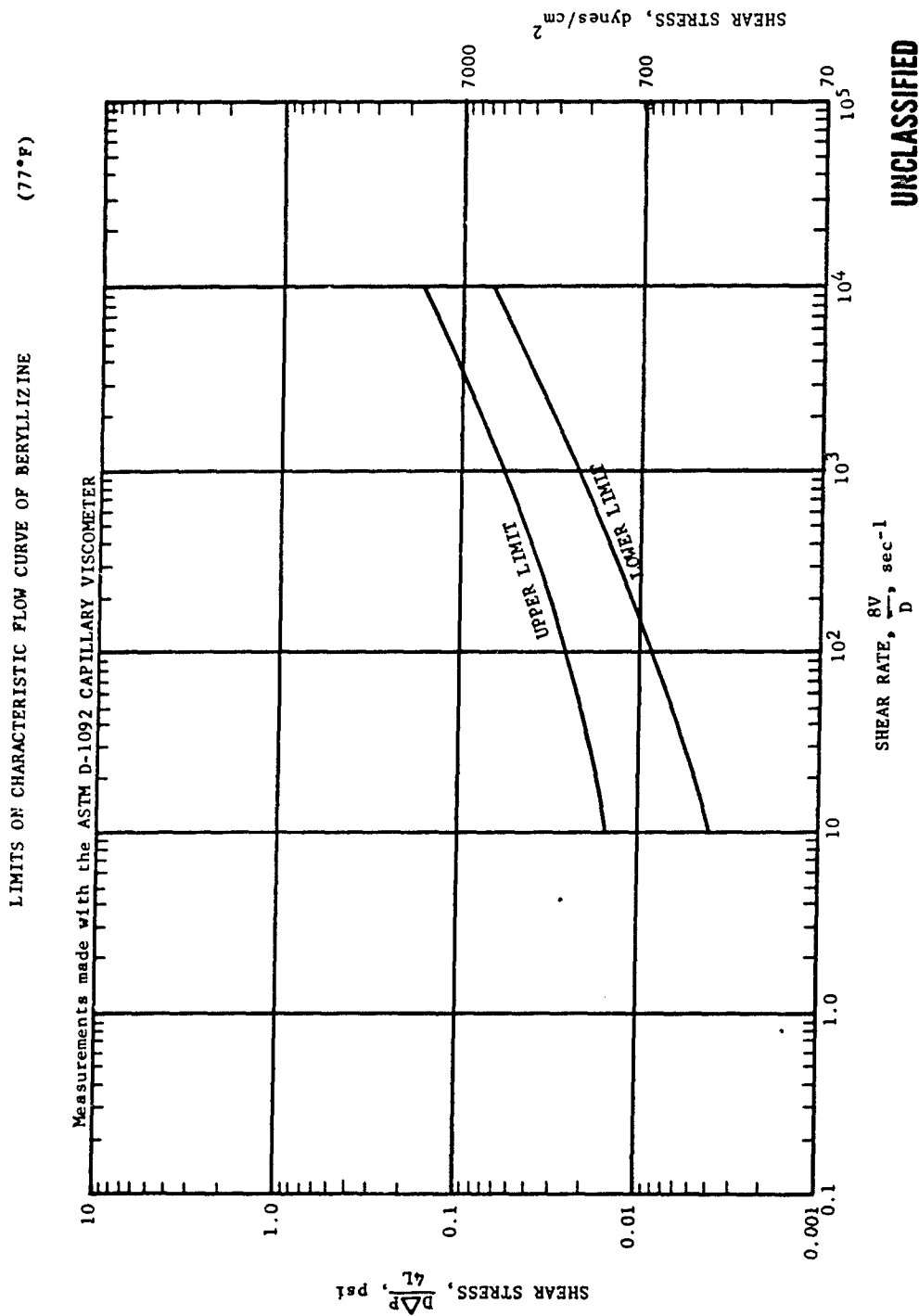


Figure 1

UNCLASSIFIED

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

(U) (g) Chamber insulation, polyurethane shield, shaped to enclose the thermostating coil and chamber

(U) (h) Dry box, nitrogen filled

(U) (i) Kimwipes, 900-L, Kimberly-Clark Corp.

(U) (j) Stoppers, rubber, size 00

#### 4.8.6.2 Calibration and constants determination.

##### (a) Pump calibration

(U) The viscometer pump flow rates, at working pressures from 0 to 100 psig, shall be determined in accordance with test method ASTM D-1092, and calibration curves shall be plotted for the 40- and 64-tooth gears, which are used on the gear reductor to drive the pump.

##### (b) Capillary constants determination.

(U) These constants are calculated and tabulated for each capillary as per the test method ASTM D-1092. The shear stress constant,  $\frac{D}{4L}$ , is calculated from measurements of the capillary diameter and length. The shear rates,  $\frac{8V}{D}$  ( $\text{sec}^{-1}$ ), are calculated for the capillaries based on measurements of the volumetric discharge rate of hydraulic oil out of the pump and the capillary diameter.

#### 4.8.6.3 Test procedure.

(U) The procedure is as specified in test method ASTM D-1092 and is supplemented by the required loading and special techniques described below in 4.8.6.3.1 through 4.8.6.3.2.

##### 4.8.6.3.1 Sample loading and chamber conditioning.

(U) (a) Prepare the chamber for sample loading by attaching a thermocouple and the first capillary (closed with mending tape on the end) into the bottom chamber cap. Then tightly screw the cap into the chamber taking note that the numbers marked on the end of the chamber and the cap correspond. Hand-tighten the top chamber-cap and purge the chamber with gaseous nitrogen. Plug the open end of the top chamber-cap with a rubber stopper.



## UNCLASSIFIED

### Report AFRPL-TR-66-230, Appendix C

#### 4.0, Quality Assurance Requirements (cont.)

(U) (b) In a nitrogen-filled dry box, remove the top chamber-cap, and carefully transfer the propellant sample from the holding vessel through an appropriate line reaching down into the bottom of the nitrogen-filled chamber. Charge the chamber from the bottom upward, and carefully avoid forming pockets in the propellant. Load the chamber to a depth of about 3-1/2 in., and remove the fill line taking care that propellant is not rubbed off onto the chamber walls. If the walls are not clean of propellant, thoroughly clean them with Kimwipes or a suitable disposable tissue. Add Nujol and fill to about 3 in. from the top of the chamber while carefully avoiding formation of bubbles. Replace the top chamber cap, and remove the loaded chamber from the dry box keeping the loaded chamber in a vertical position.

(U) (c) Select a chamber piston and remove one screw-plug and loosen the other. Insert the piston into the chamber by holding onto the wing-head of the loosened screw plug. Slowly press down the piston until the mineral oil comes through the threads. Attach the top chamber cap, and carefully fill the chamber space above the piston with hydraulic oil added through the pipe joint. Attach the chamber to the viscometer. Connect the hoses from the 77°C constant-temperature bath to the thermostating coil wrapped around the chamber and insulated with a polyurethane cover. Allow loaded chamber to temperature condition to 77°F.

#### 4.8.6.3.2 Testing.

These operations are required supplements to the procedure in test method ASTM D-1092.

(U) (a) In accordance with the ASTM procedure, circulate the hydraulic oil until all entrapped gas has been expelled utilizing the 40-tooth gear on the gear-reductor shaft to drive the gear pump. Then remove the tape over the outlet-end of the capillary and close the hydraulic oil return valve. The extruded propellant can be collected in a suitable waste container. As the flow profile is established in the capillary the gage pressure increases. When a constant pressure is attained the flow is at equilibrium. After the pressure is observed to be constant for 1 min, the gage is read and the pressure recorded. Release the pressure applied to the chamber by opening the return valve and change to the 64-tooth gear and repeat as above. Quickly exchange capillaries with no pressure applied to the chamber. Each capillary shall be tested utilizing both the 40-tooth and the 64-tooth gears. This gives data for each capillary at two different shear rates. Care should be taken in changing the capillaries to minimize exposure of the propellant in the chamber to the air.

(U) (b) After the testing is completed, the equipment shall be flushed and cleaned immediately with tap water, dried, and coated with the rust-preventative oil provided with the viscometer.

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

#### 4.8.6.4 Data reduction and curve construction.

(U) Multiply the recorded pressure drop,  $\Delta P$ , by the corresponding capillary constant to obtain the shear stress ( $\frac{\Delta P}{4L}$ , psi). Plot this measured shear stress against the known shear rate ( $\frac{8V}{D}$  sec<sup>-1</sup>) on the standard figure (Figure 1). By observation, curve-fit the data to construct the characteristic flow curve between the shear rates of 100 and 10,000 sec<sup>-1</sup>. The propellant is rejected if any part of the plotted characteristic flow curve falls out of the band delineated by the limits on Figure 1.

#### 4.8.7 Smoothness

(U) This property describes the quality of a metalized propellant with regard to deagglomeration and dispersion of the propellant components. Samples of properly prepared propellant have no lumps or specks, and exhibit smooth surfaces and a homogeneous texture. Lumps are masses of undispersed material having at least one dimension greater than 1/16 of an inch, and specks follow the same definition though they have dimensions less than 1/16 of an inch. Although propellant that is not smooth is unlikely to significantly affect performance or the operation of engine components, the presence of lumps and specks indicates poor workmanship and inferior propellant quality and is a valid basis for the rejection of the propellant.

##### 4.8.7.1 Procedure

(U) The test for smoothness shall be made by taking a 5-gm specimen from each of three samples of propellant and spreading out each specimen on a chemical watchglass with a spatula. After a rapid visual inspection is made of the fresh surface, the smear and inspection are repeated on the specimen. If lumps and/or specks are observed, the property is reported as "contains lumps" and/or "contains specks." For propellant that does not contain lumps or specks, the property is reported as "smooth."

#### 4.9 Rejection and Retest

(U) When any drum or sample of propellant fails to conform to the requirements specified herein, the entire lot represented shall be rejected. The rejected lot shall not be resubmitted until the contractor explains fully to the Government representative the cause of failure and the action taken to prevent recurrence. After correction, the quality conformance tests listed in paragraph 4.6 shall be repeated.

## UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

### 4.0, Quality Assurance Requirements (cont.)

#### 4.9.1 Preparation for delivery inspection

(U) The preservation, packaging, packing and marking for shipment and storage of the propellant shall be inspected to determine compliance with the requirements of section 5 of this specification.

### 5.0 HANDLING REQUIREMENTS

#### 5.1 Packaging

(U) The propellant shall be packaged in drums, tank cars, or tank trucks as specified by the procuring activity. All containers shall comply with the requirements of the Interstate Commerce Commission as contained in Code of Federal Regulations 49 CFR 71-90. The propellant shall be delivered in drums conforming to Interstate Commerce Commission Specification ICC-5C and in tanks conforming to ICC-103C-W. Gasket used to seal container opening shall be of polytetrafluoroethylene material to provide a tight, compatible seal. The supplier shall assure that a gasket is serviceable and furnish a new gasket when necessary. The supplier shall perform inspection on all containers provided with inspection ports. Unless leased to the Government, the supplier shall perform cleaning of contractor-owned containers to assure that these containers are free from contamination, and suitable for shipment and storage. After filling containers in accordance with Interstate Commerce Commission regulations, the space above the liquid level shall be filled with contractor-furnished nitrogen in accordance with MIL-P-27401, Type I. Containers shall not be filled with propellant exceeding 90% of capacity at 70°F (21°C). At the time of sealing, the container, the gas pressure shall be as follows: Drums, atmospheric pressure; tank trucks, approximately 8-10 psig; tank cars, approximately 10-15 psig. Unit quantities shall be as specified by contract or order.

#### 5.2 Marking

(U) In addition to any special marking required by contract or order, containers shall be marked in accordance with MIL-STD-129 including lot, batch, or control number. The nomenclature shall be as follows: Propellant, Beryllizine.

#### 5.3 Labeling

(U) Each container shall be labeled with a white label for corrosive liquid required by regulations or statutes without exception. An additional label impervious to climatic conditions shall contain the following information in red letters:

# UNCLASSIFIED

Report AFRPL-TR-66-230, Appendix C

## 4.0, Quality Assurance Requirements (cont.)

WARNING:           FLAMMABLE, TOXIC MATERIAL  
                 HAZARDOUS LIQUID AND VAPOR  
                 MAY BE HARMFUL IF ABSORBED  
                 THROUGH SKIN OR INHALED!

## 6.0   NOTES

### 6.1   Intended Use

(U)   The material covered by this specification is intended for use as a liquid fuel for liquid rocket engines.

### 6.2   Safety Precautions

#### 6.2.1   Flammability

(U)   Beryllizine propellant meeting the requirements of this specification has a flash point of about 123°F. Smoking or open flames must be prohibited where the material is being handled.

#### 6.2.2   Toxicity

##### 6.2.2.1   General.

(U)   Harmful effects may result from skin contamination and inhalation of vapors. If contamination occurs, flush with water and remove contaminated clothing. Handling must be restricted to areas so ventilated as to provide an atmosphere in which the propellant is undetectable by odor.

##### 6.2.2.2   Beryllium hazard.

The inhalation of beryllium or its compounds constitutes a serious health hazard. Two types of conditions can result from inhalation of excessive quantities of certain beryllium compounds. One, the acute condition, is associated with the inhalation of relatively high concentrations over short periods of time. The second, or chronic condition, can result from inhalation of relatively low concentrations of beryllium aerosols over longer periods of time. For recommended limits on beryllium contamination reference should be made to U.S. Atomic Energy Commission Report No. HASL-36 (1958).

#### 6.2.3   Spills

In case of spills, flush the propellant into an appropriate container or sump with copious amounts of water.

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Report AFRPL-TR-66-230

APPENDIX D

LABORATORY PROCESSING PROCEDURES FOR BERYLLIZINE (u)

Page 225

**CONFIDENTIAL**

(This page is Unclassified)

**CONFIDENTIAL**

Report AFRPL-TR-66-230

Page 226

**CONFIDENTIAL**

(This page is unclassified)

# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix D

## LABORATORY PROCESSING PROCEDURE FOR BERYLLIZINE - 21G55\*

### A. EQUIPMENT

1. (C) The 1/2-gal laboratory mixer shown in Figure 1 is used to make Beryllizine with a modification of batch size. Because of beryllium cost, a batch size of 2000 cc is utilized. Two six-blade standard turbines of 3-in. diameter are used. With the batch size of 2000 cc the two turbines are adjusted to the locations one-quarter of the batch height beneath the surface and the same distance off the bottom. For record-keeping purpose, batches made in this mixer (Figure 1) are designated LP-XX.

2. (C) A second laboratory mixer, which is a scaled-up version of the mixer in paragraph 1 above, is used to make 3-gal batches by the standard procedure. These 3-gal batches are designated by batch number, 3LP-XX.

### B. SAFETY

(U) All handling and mixing operations shall be conducted in laboratories approved by the Industrial Hygiene Office, and all procedures shall comply with established safety practices and conditions specified in "Industrial Hygiene Procedures for the Fabrication and Testing of Propellant Systems Using Beryllium," Industrial Hygiene Office, Sacramento Plant, Aerojet-General Corporation, 6 April 1965.

### C. OPERATIONS

1. (C) Charge the beryllium power (except that withheld for combining with the gelling agent) to the mixer and attach the top assembly. Reduce the pressure in the mixer to the vapor pressure of hydrazine.

2. (C) The gelling agent is weighed, combined with 3 times its weight of beryllium powder, and, after dry-blending, the combination is set aside. This mixture promotes efficient gelation of the hydrazine when used later.

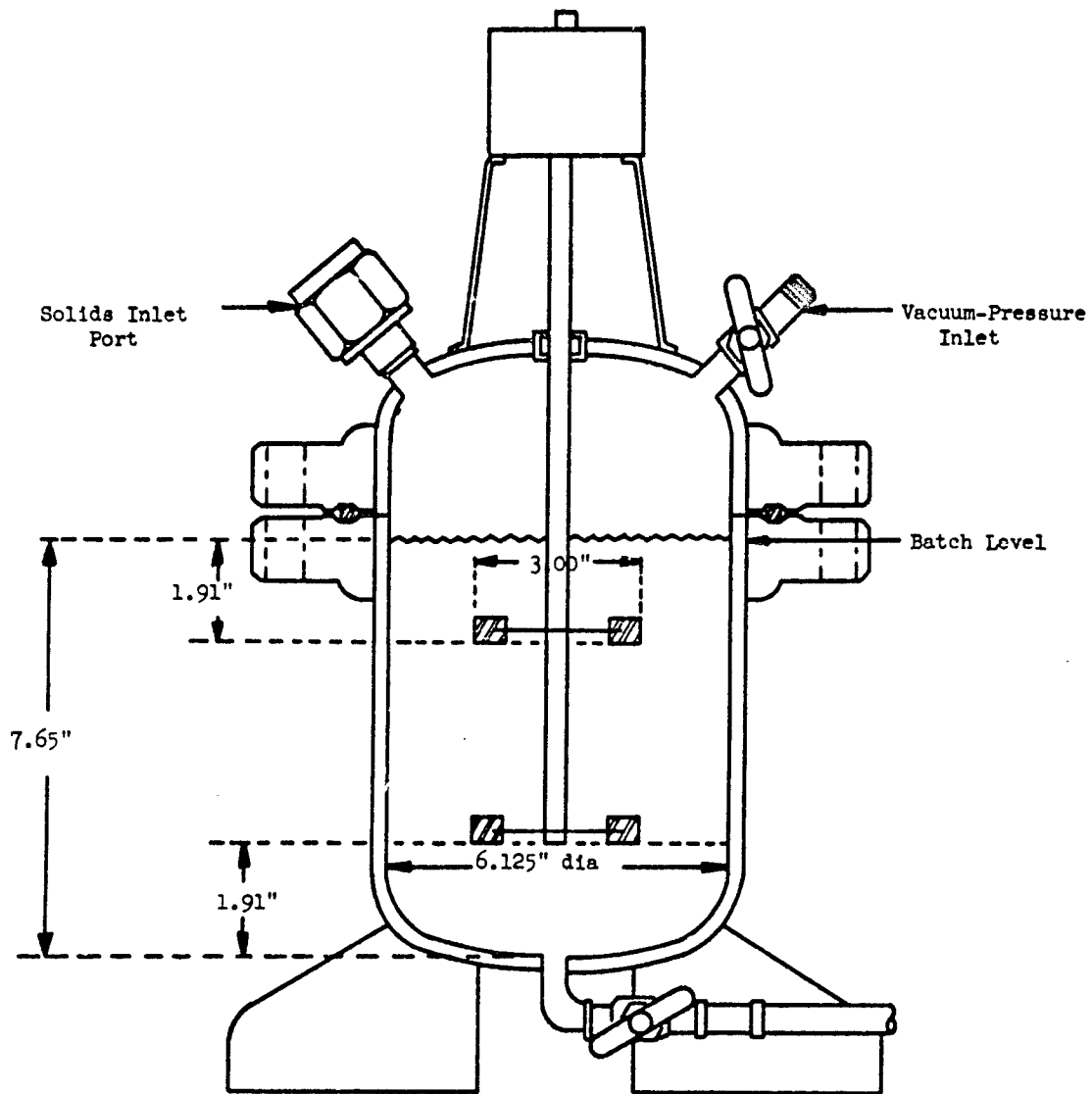
3. (C) Add the premeasured hydrazine to the mixer, and disperse the beryllium and the hydrazine for 10 min at a turbine speed of 1400 rpm while maintaining the mixer at the vapor pressure of hydrazine.

4. (C) Stop mixing and pressurize the mixer with nitrogen gas to atmosphere pressure. Open the solids-inlet port and add the preblended mixture of Carbopol-940 with beryllium powder (ratio of 1:3). The addition of gelling agent to the mixer should require a duration of 1 min while the turbines are rotated at a speed of 500 rpm. After all agent is added to

\* Beryllizine-21G55 is a fuel containing 21% of beryllium in hydrazine gelled with 0.55% of Carbopol-940, which is designated by the letter G.

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Report AFRPL-TR-66-230, Appendix D



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Figure 1 1/2-Gallon Laboratory Mixer

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(This Page is Unclassified)



# CONFIDENTIAL

Report AFRPL-TR-66-230, Appendix D

the mixer, the turbines are stopped, the inlet port is closed, and the mixer pressure is reduced to the vapor pressure of hydrazine and the vacuum system closed. The final dispersion is conducted immediately.

5. (C) The final dispersion is performed by mixing for 10 min at a turbine speed of 1200 rpm.

6. (U) Pressurize the mixer with nitrogen gas to 20-30 psig and withdraw samples.

## (C) MIXING OPERATIONS SUMMARY

TO PREPARE: Beryllizine-21 (Containing 0.55% Gelling Agent)

MATERIALS REQUIRED:	1. Commercial hydrazine	1725.9 gm
	2. Beryllium powder	462.0 gm
		(withhold 36.3 gm for combing with gelling agent)
	3. Carbopol-940	$\frac{12.1 \text{ gm}}{2200 \text{ gm (2000 cc)}}$

<u>STEP</u>	<u>OPERATIONS</u>	<u>MIXING</u> <u>Time, min</u>	<u>TURBINE</u> <u>Speed, rpm</u>
1.	Add Be to mixer.  Reduce pressure to vapor pressure of hydrazine.		
2.	Add premeasured hydrazine to mixer.  Mix.	10	1400
3.	Add gelling agent preblended with Be.  Preblend composed of 1 part Carbopol-940 and 3 parts Be.  Additional duration 1 min.	1	500
4.	Final dispersion.  Mix.	10	1200
5.	Pressurize mixer and sample.		

**CONFIDENTIAL**

Report AFRPL-TR-66-230

Page 230

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11 SUPPLEMENTARY NOTES None		12 SPONSORING MILITARY ACTIVITY Rocket Propulsion Laboratory Air Force Systems Command Edwards, California
13 ABSTRACT (C) $N_2O_4$ /Beryllizine-21(I) and 98% $H_2O_2$ /Beryllizine-28(II), were investigated in a 1000-lb thrust test assembly operated at 500-1100 psia chamber pressure to develop techniques for ignition and efficient combustion of the metal. Ignition was achieved by the use of an injector concept that involves a two-part design. Each of the two types of injectors designed for the investigations contained a central hot-core section within a second, peripheral injection area. By operating the hot-core at a high mixture ratio, the temperature of combustion of the oxidizer with the hydrazine in the fuel was high enough to ignite the suspended beryllium. The peripheral section of the injector was operated at such a mixture ratio as to produce an overall ratio favorable to high efficiency of combustion. For (I) the highest specific impulse obtained was 321 sec (86.3% of theory) at a mixture ratio of 0.71 at 1100 psia chamber pressure. In the case of (II) the highest specific impulse obtained was 344 sec (90.3% of theory) at a mixture ratio of 1.1 at 977 psia chamber pressure.  (C) The flow properties of Beryllizine-21 were also investigated. The ASTM-D-1092 capillary viscometer was used to characterize flow in .		

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Page 1

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Report AFRPL-TR-66-230

the laminar region. Alumizine-43G was also examined for comparison purposes and the data show that these two fuels have similar flow properties. When Beryllizine-21 was flowed in 1/4-in. dia. stainless steel test sections, the results were consistent with those obtained with the capillary viscometer. Flow loop tests with Beryllizine-21 in the turbulent region showed that in this case also the flow properties are nearly the same as those of Alumizine-43G when the two fuels have comparable yield stress values.

(C) The fuels used in the engineering investigations were prototype formulations made with materials and processes that were state-of-the-art at the beginning of the program. These fuels had desirable physical and rheological properties, but they were not suitable for long-term storage due to the chemical incompatibility of the components. Propellant investigations were conducted concurrently with the engineering investigations to determine the sources of incompatibility and to develop and characterize final formulations that were mechanically and chemically stable during long periods of storage. The chemical compatibility investigations showed that, in general, the purest beryllium powders were the most compatible with hydrazine. The impurities that were most suspect as sources of evolved gas were beryllium carbide and magnesium. The water-treatment of chromium-coated powders, particularly those with low contents of impurities, was shown to be the most effective of the methods investigated for the reduction of gas evolution. In addition, it was shown that Carbopol-940 used to gel the prototype formulations played an important role in the gas-producing chemical reactions. On the other hand, certain Aerojet-developed derivatives of polyacrylic acid were effective gelling agents for Beryllizine but did not affect the chemical stability of the fuel. By combining the use of the new gelling agents with the water-treatment of the beryllium powder, the formulation of chemically stable Beryllizine formulations was made possible.

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Report AFRPL-TR-66-230

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14	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
	Fuel, Rocket (U) Beryllium (U) Hydrazine (U) Gelled Fuel (U) Metalized Fuel (U) Ignition (U) Combustion (U) Nitrogen Tetroxide (U) Hydrogen Peroxide (U) Rheology of Thixotropic Fuel (U) Non-Newtonian Fluid (U) Beryllizine (U) Two-Phase Flow (U)						

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Report AFRPL-TR-66-230

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Texaco, Inc. P.O. Box 509 Beacon, New York 12508 Attn: Dr. R. E. Conary, Manager	1	The University of Utah College of Engineering 1400 E. Second South Salt Lake City, Utah 84112 Attn: Prof. M. L. Williams	1
Union Carbide Corporation 270 Park Avenue New York, New York 10017 Attn: C. R. Buell, Security Officer	1	Los Alamos Scientific Laboratory University of California P. O. Box 1663 Los Alamos, New Mexico 87544	1

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